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# STEELPLANT REFRACTORIES

*TESTING, RESEARCH AND DEVELOPMENT*

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*By*

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*SHEFFIELD*

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## FOREWORD TO THE FIRST EDITION

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**I**T IS WITH SINCERE PLEASURE that I accept the author's invitation to write a short foreword to this book, and take this opportunity of paying tribute to the energy and ability which he has applied in the organising and managing of this section of our activities. He is possessed of a facility for collaboration, as will be noted from the generous references to his colleagues and associates, which is in itself a great asset in research of any kind. This has undoubtedly contributed largely to the comprehensive character of the information contained in the book.

Refractories are a very important commodity ancillary to metallurgical and other industries and in no field of metallurgical industry has greater progress been made during the last decade than in refractories. We take great care in the designing and building of furnaces, but unfortunately the operating temperatures are often so near to the limiting useful temperature of refractories, that it is a constant source of anxiety to get the maximum output and most effective operating temperature, without deforming the vital parts of the furnace and destroying or at least reducing its effectiveness. Moreover, refractories represent one of the main individual items in steelmaking costs.

During the last few years, we have seen great advances made towards the All Basic open-hearth furnace, which should ultimately prove to be an important factor in producing better steel. One has pleasure in commending the great contribution made by pure scientists in this and other fields of refractories, with particular reference to the use of X-rays. This has added in an outstanding manner to our knowledge of the constitution of the materials involved and their changes in service. It is work of this kind, together with orthodox chemical analysis and petrological work, as well as painstaking observation in service, which have brought about the advancement to which I have referred.

I would like to add my tribute to the work of the British Refractories Research Association and to the collaboration between the manufacturers of refractories and the users largely through the Open Hearth Refractories Joint Panel (of which I have had the honour to be

Chairman since its inception) and its Sub-Committees. I regard this as an outstanding example of collaborative research in its best sense. It is impossible at the moment to recount all that has been done in the war effort, but the members have reason to be proud of their achievements.

I have no hesitation in commending this book, which blends so admirably fundamental research with practical experience.

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Chapter I

METHODS OF TESTING

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THE SLIGHTLY MALICIOUS JOY sometimes aroused by poor correlation between laboratory tests on refractories and their performance in service should not be allowed to hide the fact that brick behaviour in furnaces can be predicted far more certainly from laboratory tests than from casual examination or sales propaganda. The difficulty of achieving quantitative relationships is, as Clements rightly affirms, due in part to an over-simplified approach to the time/temperature relationship, the natural tendency being to equate high-temperature/short-time laboratory tests with low-temperature/long-time practical conditions. It is also due to the fact that bricks are usually heated in service from one side only, whereas in the laboratory they are often heated on all sides.

In spite of these limitations, laboratory testing has proved its worth in the following roles:

- (1) The checking of new deliveries, *e.g.*, to see whether a premium silica brick has in fact an alumina content or porosity in the range specified.
- (2) The evaluation of a new material prior to works trial, *e.g.*, checking a supplier's claim of increased thermal shock resistance.
- (3) The *post mortem* examination of used refractories, *e.g.*, to see whether silica bricks of a type that failed in a slag pocket arch had a low refractoriness under-load.

In addition testing plays a vital part in the development of new refractories, recourse being frequently made to standard methods to ensure that the new material is generally satisfactory and not just an improvement in some specific manner.

That testing does not have to be remarkably successful in order to pay for itself can be deduced by even casual consideration of works costs. Actual figures vary greatly from plant to plant but the following



may be taken as reasonably typical of 1956 costs on British furnaces:

	<i>Materials + Labour</i>
	<i>(per ton of steel)</i>
	<i>s. d.</i>
Basic open-hearth furnace:	
Hot metal tilter 300 ton . . . . .	8 0
Cold pig fixed 100 ton . . . . .	15 0
Acid open-hearth furnace . . . . .	17 6
Acid bessemer converter . . . . .	5 0
Basic bessemer converter . . . . .	7 0
Electric arc furnace . . . . .	17 6
Casting-pit:	
Ladle bricks . . . . .	2 0
Bottom pouring refractories . . . . .	4 0

It will be seen from the above that the refractory materials and labour costs for 1-ton of steel run at about 15s., which represents an expenditure on a 2 million ton production of £1,500,000 per annum. Even a 1 per cent. saving on this amount would be sufficient to maintain a laboratory with 5 graduates and 10 assistants—quite a substantial effort for a firm having a production of 2 million tons of steel per annum.

The demonstration of actual savings due to testing or research is always a delicate, if not a dangerous, procedure, since the credit for any improvements may well be claimed by more than one party, whilst real savings may be completely swamped by other changes in operating conditions, *e.g.*, increased driving rates. Even so it should not be difficult for a well-established laboratory to point to developments that have resulted in savings considerably in excess of those necessary to justify its existence.

In considering refractories costs, allowance must always be made for changes both in price levels and labour rates. Unless this is done it is impossible to say whether refractories are giving better or worse service than they did in the past. The alternative method, of quoting the total number of pounds of refractories used per ton of steel made, although affording a second check on cost data, is liable to be quite misleading, it being no more justifiable to add pounds of silica and chrome-magnesite brick than it is to add apples and oranges. This point is well brought out by the all-basic furnace, where the total poundage is almost inevitably lower than that for its silica equivalent, but the costs may well be higher unless compensated by higher driving rates.

Since advantage is often taken of improved refractories to obtain greater outputs, refractories costs should, where possible, be taken in conjunction with the corresponding fuel costs and output data. Thus a speeding-up of, say, 10 per cent. on an open-hearth furnace, made possible by the use of special refractories, and leading to, say, a saving of 10 per cent. in fuel, may justify an increase of as much as one third in refractories costs.

## STANDARD TEST METHODS

It is with real pleasure that we adopt in this second edition the recently established British Standard Methods of Testing Refractory Materials. These specifications (B.S.1902 : 1952), which are the result of many years of work, are reproduced by kind permission of the British Standards Institute as Appendix 20 of the present volume. They should logically appear in the present chapter and have only been placed in the Appendix for the convenience of the regular user. Some day it may be possible to replace them again, this time by International Standards, but meantime all references to test data relate to B.S.I. Methods unless other procedures, *e.g.*, the A.S.T.M. Standards (1952) or the German D.I.N. documents are quoted. Fortunately the effect on old test data of this change to B.S.I. Standards is relatively small and older data have for the most part been retained.

Before proceeding to discuss individual tests it is perhaps wise to stress the desirability of viewing refractory test data as a whole. To say, for example, that one magnesite brick is better than another merely because it has a lower porosity or a higher cold crushing strength, whilst ignoring other features, *e.g.*, a rise in the lime content or a suspiciously low thermal shock resistance, can be distinctly dangerous. The safer approach is that adopted in interviewing a candidate for a new job, his suitability not being determined solely in terms of his academic qualifications and service experience but rather on the overall impression created on the interviewer, including the comments made by others with whom he has previously worked.

## CHEMICAL ANALYSIS

The new British Standard Methods of Testing Refractory Materials include the procedure to be adopted when analysing silica and siliceous refractories, firebricks and aluminous refractories, whilst the new A.S.T.M. (1952) Standards go further and include methods for magnesite, dolomite, chrome ore and chrome refractories. Although chemical analysis can be of great value and may even be crucial, it is

more often used as a method of classifying materials. It shows for example whether a brick is a typical silica, magnesite, dolomite, chrome or fireclay product, or, say, a composite brick, such as chrome-magnesite. If it is composite then the proportion of the main constituents can be estimated. With certain proprietary materials, such as castables, it provides the user with background information that indicates whether the material is suitable for jobs other than those specified. It must be stressed, however, that chemical analysis only provides the overall result, telling nothing about distribution. Thus an alumina content of 60 per cent. can be obtained by the use of bauxite, sillimanite or andalusite, but the physical properties of products having similar chemical analyses may be very different. In general it may be said that chemical analysis is only important in as much as it affects physical properties, such as refractoriness-under-load, which can in any case be better determined by direct testing. In some refractories, *e.g.*, silica bricks, even small amounts of impurities, such as alumina or alkalis, can have a marked effect, and here chemical analyses may be of very direct importance. Similarly magnesite for use in special chrome-magnesite bricks may be required to have a lime content of less than, say, 2 per cent, whilst with fireclay bricks the maximum content of iron oxide and alkalis are sometimes stated.

Where large numbers of chemical analyses have to be carried out, the possibility of using rapid methods, such as the spectrograph or the flame-photometer, should be considered. It has, for example, been shown that considerable time can be saved by doing alumina contents in silica bricks by spectroscopic means and the alkali content of clays by means of the flame-photometer, in which a solution is sprayed into a controlled flame and the amount of radiation in the appropriate wavelength determined.

#### POROSITY AND DENSITY

When faced with a new refractory problem and baffled as to where to begin, it is usually best to do a porosity. The results obtained often provide a clue as to the next step; they always provide useful background; and the test is in any case simple, quick and cheap. As an illustration of the first point may be quoted the results obtained on bricks that failed mysteriously in a boiler arch: the porosity data were quite normal but the cutting of the testpiece revealed the presence of serious making faults, subsequently found to be present in almost the whole delivery. The replacement of this refractory by a chemically similar but better manufactured product provided a complete solution.

The British Standard Method, set out in Appendix 20, which involves the soaking of the brick in water, always yields three figures, viz.: the apparent porosity  $P_a$ , the bulk density  $S_a$  and the apparent solid density  $S_{as}$ —previously referred to as the apparent specific gravity. A glance at these three figures, which are interrelated in the manner shown by the equation

$$P_a = 100 \left( 1 - \frac{S_a}{S_{as}} \right) \text{ per cent.}$$

can be quite revealing. The figures 26.5 per cent., 1.75 g.p.ml. and 2.39 g.p.ml., obtained on a silica brick, immediately suggest a rather porous product that is unlikely to be volume stable on refiring, the true specific gravity probably being within 0.1 of the apparent solid density 2.39 and, therefore, indicating a brick containing substantial amounts of raw quartz. Microscopic examination of such a brick showed a quartz content of about 30 per cent., while on refiring the brick showed a permanent linear change of +1.7 per cent. after 2 hours at 1500°C. With most refractory bricks the porosity will be found to lie between 20 and 30 per cent., principally because most manufacturers can readily produce porosities as low as 30 per cent. but anything less than 20 per cent. is increasingly difficult and expensive. In some cases, however, e.g., mould top bricks, the porosity may be deliberately kept high with a view to producing low conductivity and high thermal shock resistance. During recent years an increasing number of bricks have fallen in the less than 20 per cent. porosity class, the demand for such materials having increased greatly as a result of the repeated demonstration that low porosity bricks give better service, e.g., in open-hearth furnace roofs, or in the linings of metal mixers, and that given the necessary control in manufacture such bricks do not suffer from low thermal shock resistance. A similar demand is being met with ladle bricks and with checker bricks, where the denser product is found to give a longer life. Low porosity reaches its ultimate in electro-cast materials, the extended use of which is only restrained by their abnormally high initial cost. If "blow-holes", such as are also found in metal castings, are ignored, bricks of this type have virtually zero porosity.

Since apparent porosity can give a false impression of density where a brick contains a large proportion of closed pores, the alternative method of specifying bulk density may be desirable. This can be obtained by the porosity test, but the alternative method of measuring a number of bricks and dividing their weight by their volume is sometimes preferred. Details of a suitable procedure are set out in A.S.T.M. C. 134-41.

The apparent solid density, in addition to indicating the degree of firing with silica bricks, can be of use with other products, particularly

fireclay, in that the difference between this figure and the true specific gravity provides an indication of the amount of closed pores present in the brick.

### TRUE SPECIFIC GRAVITY

Both the British and American test methods employ a special specific gravity bottle (or pycnometer) for referee work, but for routine determinations a flask of the Rees-Hugill type, such as is described in British Standard Test No. 3, section B, is more rapid and sufficiently accurate. The procedure adopted is a very simple one, the flask (fig. 1, p. 9) being filled to a given level with xylene and the increase in volume which occurs noted when 100 g. of suitably ground and dry material are poured into it.

Various suggestions have recently been made for the improvement of such flasks. These are described in the Tentative Standard CT/LBC 4848. Rees-Hugill type flasks have also been used for the specific gravity of other materials, *e.g.*, magnesite, but are only of real value where a large number of routine measurements are to be made.

The value of specific gravity control in showing not only the average degree of conversion in silica bricks but also the spread is well illustrated by fig. 2, which shows tests made on deliveries from 3 different suppliers. When the data so obtained are expressed in histogram form (*see* Statistical Section at the end of this chapter), it becomes quite clear for example that manufacturers A and B are producing similarly converted products, but that the material produced by manufacturer C shows an extremely wide spread, which would be expected to yield in service not only a greater after-expansion but also a much more serious point to point variation. Whether a low specific gravity

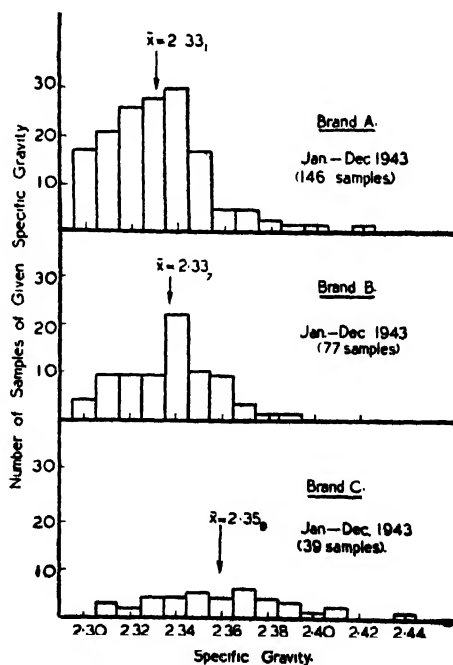


Fig. 2. Histograms showing variations in specific gravity of three brands of silica brick.

is desirable is a separate subject that will be discussed later, but what is clear is that given a specification adequate control can only be achieved by frequent testing and comparison of the results with a standard previously agreed between manufacturer and user. Thus it has been agreed that deliveries of roof bricks from one silica brickworks to one steel company shall show an average true specific gravity of less than 2.365 on 4 bricks taken at random from any delivery.

#### PERMEABILITY TO AIR

It is more difficult to justify a permeability than a porosity test but since the test (B.S.I. Standard No. 4—*see also* fig. 3, p. 9), is comparatively simple and contributes an additional feature to the background picture, it is generally worth carrying out. It has, for example, been observed that de-airing, which is generally agreed to increase durability in, say, casting-pit refractories, has a much more marked effect on permeability than on porosity, presumably because a small decrease in porosity may lead to the closing of previously open channel-type pores. Certain workers, *e.g.*, Massengale, have attempted to correlate permeability with the other properties of refractories but have not discovered any close relationship except between low permeability and high resistance to attack by molten glass.

#### REFRACTORINESS

The refractoriness of a material as determined by pyrometric cone method should not be confused with the true melting point, since the cone test is really a small refractoriness-under-load test in which the load is that of the cone itself. In addition there is a time factor involved, which probably explains why cone refractoriness figures for silica brick as high as 1730°C. are frequently quoted although this is about 20°C. above the melting point of the pure material. Where true melting points are required refined techniques, such as the heating and quenching methods employed at the Geophysical Laboratory in Washington, are recommended.

Refractories tests are particularly useful with bricks in the aluminosilicate range, the results obtained being in fair agreement with the alumina-silica system. With silica bricks, and still more with basic bricks, the refractoriness-under-load results are of more significance, basic bricks rarely failing through direct melting, but often collapsing at temperatures well below the melting point due to softening of the bond. Refractoriness tests can also be of interest on used brick samples. It has been shown for example that the grey zone on the underside of an open

hearth furnace roof has a cone melting point of the order of  $1680^{\circ}\text{C}.$ , which is very close to the temperature at which roofs begin to run seriously. That other factors are, however, equally important would appear to be supported by the practical observation that particularly hot casts of steel are preferably made in a furnace at least 3 weeks old, although the cone melting point of the underside of the roof is probably  $40^{\circ}\text{C}.$  below that of the unused brick.

In considering refractoriness data it should always be remembered that the standard test specifies an oxidising atmosphere, whereas in practice reducing gases are often present and may cause a dramatic drop in the melting point of used bricks containing substantial quantities of iron oxide. Attention should also be drawn to the fact that the British and American refractoriness tests are very different, both as regards the procedure and the nature of the cones used. The comparative end points for the latter are given in Appendix 6a, which shows, for example, that the much quoted "cone 30" has an end point of  $1650^{\circ}\text{C}.$  in the American scale, but  $1670^{\circ}\text{C}.$  in the Seger scale.

Other methods of determining refractoriness are employed, *e.g.*, the Holdcroft's thermoscope bars listed in Appendix 6b, but for steelplant use the cone test, now frequently supplemented by rare metal thermocouple readings, is fairly standard.

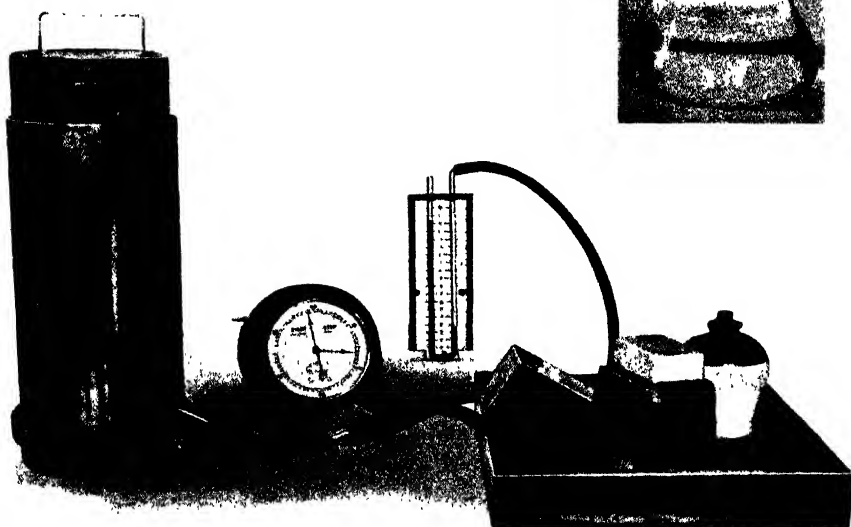
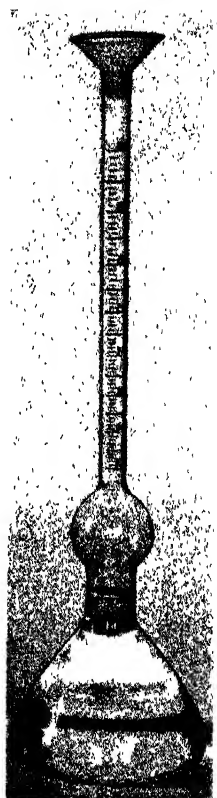
#### REFRACTORINESS-UNDER-LOAD

Although the methods to be used in carrying out refractoriness-under-load tests (*see* fig. 4, p. 10) are still in process of revision, there is no doubt that the results obtained even by the present methods are of real value. Indeed they may be said to have a double importance, in that they indicate:

- (a) The probable behaviour of refractory materials under load, *e.g.*, in furnace arches.
- (b) The general quality of the fired product.

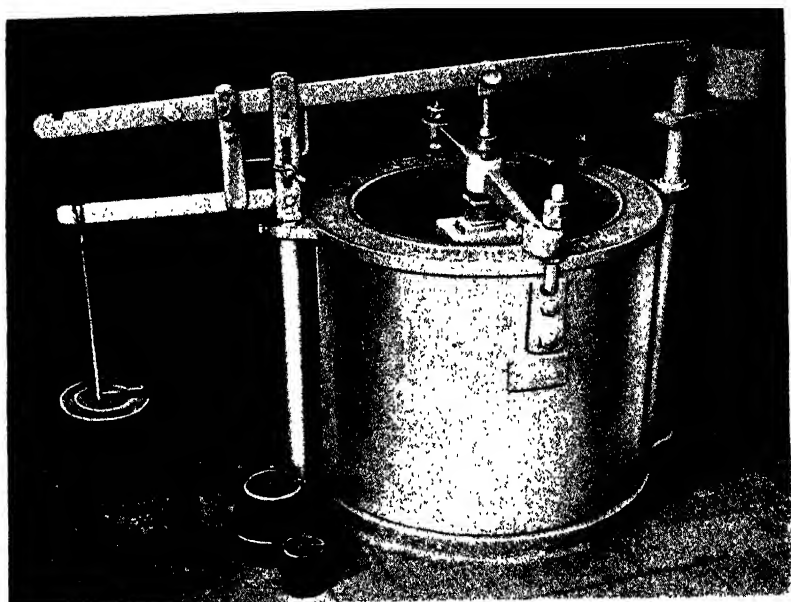
Two main types of test are employed, *viz.*, the rising temperature method, which can be used for example to show the presence of excessive fluxes or inadequate firing, and the maintained temperature test, which measures the ability of the material to carry a given load over short periods. The procedures to be adopted in these tests are set down in B.S.I. Standard Method No. 6, from which it will be seen that for silica bricks, whose load bearing characteristics are usually excellent, a 50 lb. per sq. in. (3.5 kg. per sq. cm.) load is specified, while for other materials the standard load is 28 lb. per sq. in. (2 kg. per sq. cm.). For comparison it is interesting to know that the calculated stress in a typical open-hearth furnace roof is about 25 lb. per sq. in., though pressure cell

*Fig. 1. Rees-Hugill flask for routine determination of specific gravity of silica bricks.*



*Fig. 3. Apparatus for determining permeability to air of refractory materials and insulation. Gasometer on left, test-piece sealed into holder with plasticine on right. Mercury seal still to be made.*





*Fig. 4. Refractoriness-under-load apparatus- Hirsch type carbon granule furnace.*



*Fig. 5. Thermal shock test-pieces showing typical failure (left to right) on stabilised dolomite, silica, fireclay and chrome-magnesite test-pieces.*

measurements show the distribution to be uneven and local loadings of 100 lb. per sq. in. or more to occur. With vertical walls the dead loading rarely reaches even 25 lb. per sq. in., though excessive loads may occur due to nipping where insufficient expansion allowance has been made.

Until comparatively recently British technologists normally used the rising temperature test but the observation that many bricks would stand quite a high load for a short time and yet be unserviceable at temperatures appreciably below the fail point has led to the increasing use of the maintained temperature test, particularly for materials used in arch construction. Indeed, the most recent trend goes further and employs long term creep tests, similar to those used by metallurgists but carried out not only in tension but also in torsion and compression. It has as a result been demonstrated that bricks having fail points as high as 1700°C. in the rising temperature test may continue to creep at 1400°C. under a similar load until failure occurs. It appears from such observations that in many arch structures the load is soon transferred to the cooler part of the brick, the effective thickness of the arch being only, say, threequarters of the brick length employed.

Even these improved methods suffer from the disadvantage that a test-piece is maintained at one temperature, whereas in practice a steep temperature gradient from hot to cool face exists. The difficulties of simulating such conditions in the laboratory are so great that recourse is now being had to full-scale test-rigs for the study of the refractoriness-under-load of such special materials. Those who wish to go into the details of refractoriness-under-load tests are referred to the recent surveys by F. Clements and L. Halm, where the methods employed in different countries are discussed. Most of the test-pieces used in Europe are quite small, but the A.S.T.M. (*see* C16-49) still specifies the use of a full 9 in.  $\times$  4½ in.  $\times$  2½ in. brick, or specimens of this size cut from larger blocks. Many arguments can be produced in favour of using a full brick, *e.g.*, the direct demonstration of manufacturing faults, but it is perhaps worth noting that the usefulness of the test depends not only on the precise relevance of the data collected but also on the amount of data, which is likely to be much reduced if full bricks rather than small test-pieces are employed. It is also perhaps relevant to point out that in the A.S.T.M. test the brick is stressed on end, a condition very rarely observed under service conditions.

Certain refractories, *e.g.*, high grade fireclay bricks, behave quite satisfactorily when used even as thin arches, but if insulated are liable to collapse due to the gradual softening of the material. Refractoriness-under-load tests enable some indication to be given of the extent to which insulating materials can be used, demonstrating for example that

where high temperatures and insulation are involved bricks of much higher alumina content, *e.g.*, sillimanite, may be desirable.

The increasing use of unfired refractories, mostly of the basic type and involving metal reinforcing or jackets, presents the refractories technologist with a new problem to which there is no easy answer. What is clear is that within a day of going into service such bricks must vary greatly in properties from the hot to the cool face and that a refractoriness-under-load test carried out on an unused brick probably tells very little about behaviour under service conditions.

#### PERMANENT LINEAR CHANGE ON REHEATING

This property, often referred to as after-contraction or expansion, is of direct importance, particularly with basic bricks, since it may reveal soft-firing either of clinker or brick, or the presence of an undue amount of flux. With stabilised dolomite bricks it provides an indirect measure of probable stability in storage—hard firing being the best guarantee of subsequent freedom from hydration troubles in bricks having the correct chemical composition.

Chrome-magnesite bricks rarely show much change on reheating, indeed the mould sizes used in the work are frequently those required in the final product. With silica bricks difficulty is often experienced in obtaining reproducible results, mainly because soft-fired material is likely to show crozzle cracking on refiring as well as surface roughening due to the protrusion of large grains in the fired test-pieces.

The greatest difficulty, however, lies with fireclay bricks, which after years of study must still be considered as being in the "department of utter confusion". Clements, who has spent several years in studying this subject, still considers the present position unsatisfactory, and points out that contraction and expansion can occur consecutively in the course of the same test—the one effect tending to mask the other. Furthermore the change in dimensions varies so much with the test temperature that entirely different results may be obtained according to the precise temperature chosen. Finally test-pieces cut from the same brick can show substantial differences, partly it is assumed due to non-homogeneity of the material, but partly also because of comparatively small point to point differences in test conditions.

The test period in the British specification is only 2 hours, but in the American, where a full brick is employed, a test time of up to 5 hours is employed. In spite of all these difficulties and differences useful results are being obtained by the test and there is general agreement on certain points, *e.g.*, medium alumina firebricks containing a large

amount of alkali show substantial shrinkages, particularly if soft-fired, whereas other less refractory materials show a marked bloat, due apparently to the expansion of gases in the sealed pores. As will be seen from Chapter XIV, bricks of this type are frequently specified for ladle linings.

#### COLD CRUSHING STRENGTH

It is very rare that refractories fail due to dead loading in the cold condition. Tests of this property are therefore generally carried out with a view to checking indirectly some other property, *e.g.*, abrasion resistance. It has for example been shown that chrome-magnesite bricks having crushing strengths of less than 1000 lb. per sq. in. are liable to lose corners and edges merely by being transported from place to place in a melting shop, whilst such relatively weak bricks may even fracture during handling and thus result in serious wastage. The indirect value of cold crushing strength data lies in examples such as those given below:

- (1) The degree of firing of stabilised dolomite bricks, which as already mentioned controls at least in part their stability in storage, can be conveniently assessed by cold crushing strength tests.
- (2) Low cold crushing strengths in fireclay bricks are generally an indication of under-firing and frequently suggest the desirability of further checks, *e.g.*, of permanent linear change on refiring.
- (3) Extremely low cold crushing strengths are liable to result from the use in chrome-magnesite bricks of chrome ores susceptible to growth in alternating oxidising and reducing atmospheres. Since it is found that repeated firing of such bricks leads to further deterioration similar difficulties may well occur in service.

Both the A.S.T.M. test and the new British Standard permit the use of full 9 in. bricks loaded on end, but the British specification also permits the use of 3 in. cubes. Both test-piece size and direction of pressing should always be indicated in the test report, since the presence of even slight laminations in the brick may have a substantial effect on the result obtained. In all such work multiple testing is essential to take care of the wide spread in crushing strengths obtained on bricks from a given batch.

#### REVERSIBLE THERMAL EXPANSION

This property is of direct importance, in that adequate expansion allowance should be made in furnace structures, but it is equally

important in that it shows up peculiarities in expansion, *e.g.*, the cristobalite change in a silica brick, which may have a marked effect on thermal shock resistance. An American steelplant man once stated that ceramic engineers straight from college usually recommended the theoretical expansion allowance, whereas in practice none was required. The truth would appear to be somewhere in between: a figure of roughly one-half the theoretical expansion proving most useful. This difference between simple theory and actual practice is probably due in the main to the take-up which occurs in cement joints, or even in dry joints, between the bricks. Rigby, for example, has shown that if a brick is cut in half and the two rough ends pressed together, a substantial contraction can occur before any fracture of the brick takes place. This accommodation is presumably due to the crushing of surface roughnesses under loads well below the crushing strength of the brick. Measurements made on all-basic furnace arches also indicate that only one-third to one-half of the expansion in an arch built of chromemagnesite bricks separated by steel plates is actually transmitted to the suspension mechanism.

This is clearly a field in which the advice of the experienced builder must take precedence over theory, though many anomalies can be explained if due account is taken of temperature gradients between the inside and outside of the roof and permanent expansion or contraction effects, such as for example the growth on soft-fired silica bricks, which as every builder knows can lead to an abnormal rise. Incidentally such movements are shown up in thermal expansion tests if the furnace is heated to a temperature in excess of that used for the firing of the bricks.

Where abnormally rapid expansions are found due to inversion from one crystal form to another, *e.g.*, the cristobalite and quartz changes in silica, and the very peculiar inversions observed with zirconia, special precautions must be taken in service to avoid cracking on heating-up. The ideal procedure has still to be worked out but it is generally agreed that damage can be greatly reduced by specifying a maximum safe rate of heating through the critical range, which in silica means up to, say, 600°C. Incidentally the fact that the brick has been through these changes once is no excuse for more rapid rates on subsequent reheating or cooling, though bricks do sometimes survive such treatment, probably because they have become fused or slagged into a monolithic structure and though cracked do not actually break away.

**RESISTANCE OF A REFRACTORY MATERIAL TO THE  
DISINTEGRATING EFFECT OF CARBON MONOXIDE**

This Standard has been reproduced in Appendix 20 but more for completeness than for any relevance to the steelplant operator. It is true that when fireclay bricks containing iron spots are exposed to carbon monoxide at temperatures around 450°C. they frequently disintegrate due to carbon deposition. Such conditions are comparatively common in blast-furnaces but extremely rare in steelplants.

**RESISTANCE TO THERMAL SHOCK**

The test for this property, as described in the Appendix to the B.S.I. specifications, and illustrated in fig. 5, p. 10, although wide open to criticism nevertheless provides a considerable amount of useful background information. It is unlikely, for example, to be an accident that bricks used in positions where thermal shock is severe, *e.g.*, open-hearth furnace doors, generally show a high thermal shock resistance in the small prism test. Incidentally it may be of interest to put on record the origin of the mystic "30+". In the early days of the chrome-magnesite brick valid claims of high thermal shock resistance were made by certain manufacturers for their product. After using test piece sizes varying from standard squares to 1 cm. cubes, it was found that the 3 in. × 2 in. × 2 in. test piece, which could readily be cut from a 3 in. square, was most convenient. Since the best chrome-magnesite bricks available were found to show their first cracks at about 30 reversals a figure of "30+" was adopted as a standard and was incidentally soon achieved by other manufacturers.

The more recent test described by Howie and subsequently by other workers, in which test pieces or full bricks are heated from one end by means of an electric hot plate and the rate of heating required to cause cracking determined, is undoubtedly closer to service conditions. Here again test piece size is found to have a marked effect, the differences observed between small prisms of different qualities almost disappearing when full size roof bricks are employed.

The nearest approximation to service is of course provided by the A.S.T.M. panel spalling test, whose wider adoption has only been prevented by the laborious operations involved and the inevitably high cost of obtaining test data. If a similar type of panel test could be developed using a much smaller number of bricks and occupying a shorter test period it would doubtless achieve a wider popularity.

## MICROSCOPICAL EXAMINATION

So much progress has been made in recent years in the examination of polished sections of refractory material by reflected light, that it must be conceded that there are now three rather than two principal methods of studying refractories under the microscope, *viz.*, the powder, thin section and reflected light techniques.

## POWDER METHOD

The sample is first ground in an agate mortar until it ceases to feel gritty (approximately 200 mesh) and a small amount then transferred to a microscope slide. If the probable nature of the material is known, a liquid of approximately the same refractive index is placed on the powder and the whole covered with a thin cover glass. If the mineral happens to have the same refractive index as the liquid, it will (unless coloured) be almost invisible. If visible then whether it is higher or lower in refractive index can be readily determined by noting the direction of movement of the Becke line when the objective is moved. This line takes the form of a halo of light around the edge of the particle, which expands out into the liquid or contracts into the mineral when the objective is moved towards the stage according to whether the mineral has a greater or less refractive index than the medium.

Liquids having a wide range of refractive indices, usually from 1.45 to 1.74, but going as high as 2.00 if desired, may be prepared by mixing a series of liquids of known refractive index, followed by calibration with a refractometer. Alternatively such liquids can be purchased ready mixed and arranged in a continuous series with 0.01 intervals. Where this latter course is adopted it should be remembered that certain of the liquids are liable to change their refractive index with time and that periodic checking of the series is therefore necessary.

Powder mounts of the same type can be used for the determination of other properties, *e.g.*, birefringence, or whether the mineral is uniaxial or biaxial, being more suitable for this purpose than a thin section. The optical properties of a series of minerals associated with refractories and slags are given in Appendix 10.

A major advantage of the powder method is the speed with which it can be applied to estimate, say, the amount of raw quartz in fireclay or the degree of firing of a silica brick. It can also give invaluable information of the nature of powders, such as cements for induction furnace linings.

## THIN SECTION METHOD

With this technique, in which a section, usually about 1 in.  $\times$   $\frac{1}{4}$  in. and approximately 1/1000 inch thick, is cut through the sample, the results obtained depend so much on the finesse of the operator that practical training is to say the least highly desirable. Those unable to obtain such training can, however, make good progress by following the precise descriptions given for example in G. R. Rigby's excellent book on *Thin Section Mineralogy of Ceramic Materials*. The general procedure is quite simple, the first stage being to grind a flat face on a slice or chip from the brick by means of a lap and various grades of silicon carbide grit, *e.g.*, 80, 220, 400 and 500. The clean dry face is then attached to a microscope slide by means of Canada balsam and the other surface ground away, particular care being given in the final stages in order to avoid tearing, a risk considerably reduced by previous impregnation of the sample with resin or some suitable plastic. In general the aim should be to get the sample as thin as possible, the end point being determined by the stage at which the edges of the section begin to break up. The great advantage of this method is that it reveals the constituent phases in their original locations. Something of the detail available is shown by figs. 6 and 7, p. 19, the former showing cleavage in a sample of electrically fused magnesia, and the latter the amount and distribution of serpentine in a sample of chromite. Thin sections are also extremely useful in showing up the attack of molten slags on refractories, frequently indicating some constituent as more susceptible than others.

## POLISHED SECTION METHOD

This method, although far from new to metallurgists and cement workers, has only recently been employed at all widely by ceramists. As far back as 1924 Andersen and others showed its utility, *e.g.*, in studying examples of magnesia refractory samples from open-hearth furnace bottoms, and more recently Schouten has described its application to a wide range of products. In an ingenious diagram (fig. 8) Schouten demonstrates by means of a hypothetical thin section, the advantages to be gained by the use of the reflecting microscope. Figs. 9, 10 and 11, p. 20, reproduced from his recent paper, show both the wealth of detail obtainable and the extraordinary contrast in appearance when transmitted and reflected light are used.

It must not be assumed, however, that the polished section method will replace the thin section, since the identification of minerals under reflected light largely depends on experience gained in thin section studies.



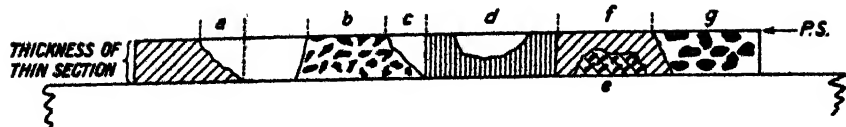


Fig. 8. Edgewise view of a hypothetical thin section. In transmitted light, section (a) will give only an approximation of the actual structure. Section (b) will appear opaque because of the presence of numerous opaque inclusions, while the apparent transparency of (c) will increase from left to right. The pit in the surface at (d), will not be directly observed. The small, opaque inclusions at (e) may appear to be in the mineral (f). With two transparent minerals finely intergrown (as at (g)) the optical properties of neither can be accurately determined. P.S. indicates polished surface. (After Schouten.)

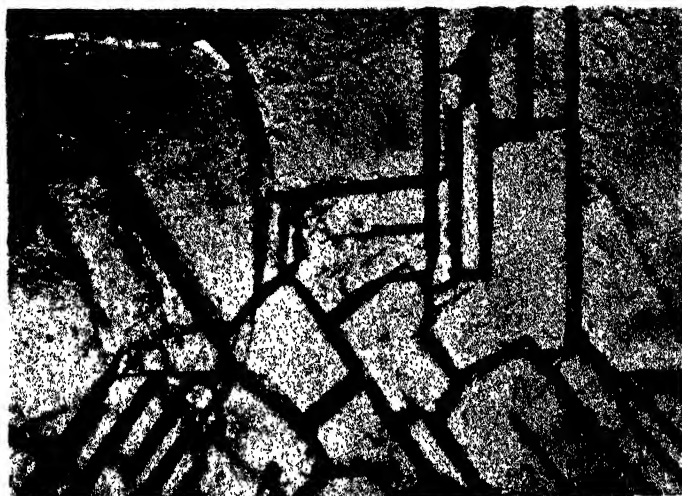
To the trained petrologist the microscope is such a powerful tool that he may be inclined to make recommendations without recourse to standard tests. This procedure is, however, far from safe, since the microscope merely reveals the type, size and distribution of the constituents, and confirmation of the probable effect of these on physical properties should, wherever possible, be obtained by direct tests.

As examples of the value of the petrological microscope may be given the ease with which an estimate of residual quartz can be given in a silica brick, or statements made regarding the nature of the raw material employed in the manufacture of basic refractories. To the trained eye, for example, there are characteristic differences between different types of magnesite and chromite, though again other methods, such as chemical analysis, or X-ray examination, may be required to supplement the evidence. With chrome ore much stress is now laid on the presence or absence of internal fissuring and inclusions, and with chrome-magnesite bricks on the proportions and size ranges of the principal constituents, including the silicate bond.

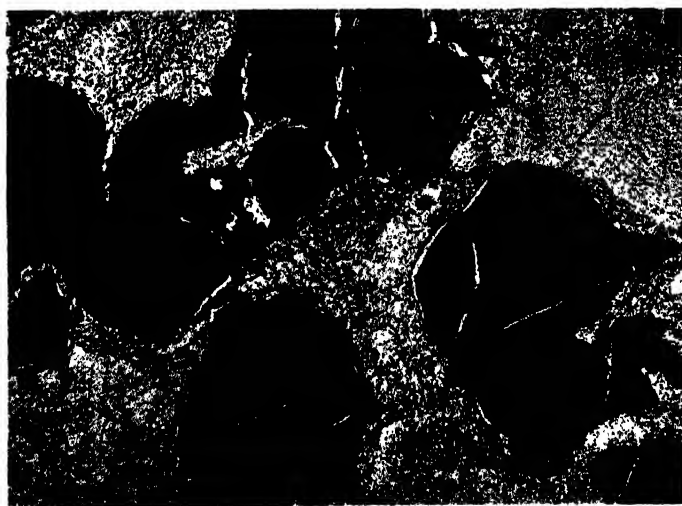
These techniques are also most useful in *post mortem* examination of refractories, often enabling the cause of some particular trouble, for example slag absorption, causing loosening of the structure, or transfer of fluxes from one brick to another, to be stated. Furthermore the picture revealed frequently suggests a possible solution to the problem. It was, for example, the observation that attack by iron oxide rich slags on Grecian magnesite was largely limited to the surface (see fig. 76, p.155, and fig. 78, p.156) that led to its extensive use in induction furnace linings.

## X-RAY EXAMINATION

The constitution and crystal size of the minerals present in a refractory can often be determined quite rapidly by X-ray examination. Such data



*Fig. 6. Electrically fused magnesia showing marked cubic cleavage. Ordinary light  $\times 60$ .*



*Fig. 7. Chromite (black) with interstitial serpentine. Ordinary light  $\times 40$ .*



*Fig. 9. Silica brick after open-hearth use. Dark-gray grains are cristobalite, light-gray interstitial growth consists of pseudowollastonite and a similar iron-bearing silicate. Almost white grains are hematite. Reflected light;  $\times 60$  (after Schouten).*



*Figs. 10 and 11. Same area of a Corhart block, in transmitted light (left) and reflected light (right). Minerals present and their structural relations cannot be clearly distinguished in transmitted light. In reflected light, gray (with relief at left edge and irregular grains at right) is corundum. Very light gray (lath-shaped dendrite) is spinel. Enclosing the spinel are mullite (short columns or rhombs) and darker-gray alumina-silica glass. Both enlargements  $\times 450$  (after Schouten).*

add both to the amount and certainty of information obtained with the microscope and frequently helps to explain the properties observed in more standard tests. The examination of a soft-fired fireclay brick under a microscope does not at normal magnifications reveal the presence of mullite, but X-ray photographs may show its presence and in addition the lines of the cristobalite and residual quartz. With unusually soft-fired material, *e.g.*, the rod covers used in ladles, another mineral, *viz.*, gamma alumina, may be found. This is a break down product from the original clay, which is reabsorbed to form mullite on harder firing. So close is the connection between the amounts of these constituents in a particular type of material and the firing temperatures, that once suitable standards have been established the final kiln temperatures can often be estimated to within, say, 50°C. This application of the X-ray technique provided a quick explanation of a serious case of steel contamination by spalled mould top bricks. A comparison of the X-ray patterns of the spalled material with those of earlier and quite satisfactory samples showed that the firing temperature employed had been raised from 1250°C. to about 1400°C., which in turn had led to a marked drop in thermal shock resistance. As will be shown in later chapters, X-ray examination has also played a major role in the development of new refractories, *e.g.*, stabilised dolomite, and also added much to the certainty of phase diagram data.

## CONSTITUTION

### (a) General

Those who wish to understand the fundamental principles behind X-ray crystallography are advised to read the textbooks listed at the end of the present chapter. For the present purpose, however, it is sufficient to know that each crystalline form has its own diffraction pattern (*see* fig. 12, page 25), which is as certainly associated with the mineral as a finger print with the criminal. Fundamental consideration of such patterns enables the actual crystal structure to be deduced, but for most purposes it is sufficient to identify the mineral and get an impression of its crystal size.

Since most refractories consist of small crystal aggregates, the powder method, originally devised by Debye-Scherrer and Hull, is most convenient. The sort of results obtained are shown in fig. 12 where it will be seen for example that the three main forms of silica—quartz, cristobalite and tridymite—although chemically identical have entirely different crystal structures. Quartz can be easily identified under the microscope, but cristobalite and tridymite, if

poorly crystalline, may easily be confused. The striking difference between the patterns given by magnesia, alumina and quartz, do not require emphasis, nor should the strong resemblance between the patterns for the ordinary magnesium aluminate spinel and the chrome ore sample. It is also interesting to note that mullite and forsterite have entirely different patterns from their oxide constituents. It is differences such as these which enables reactions between oxides to be followed, the original phases gradually disappearing and the new phase becoming dominant.

*(b) Solid solution effects*

The similarity in pattern (shown in fig. 12) between magnesium aluminate and chrome ore, is just one example of the fact that similar arrangements of atoms in the lattice also have similar diffraction patterns. In some cases, as with the spinels, the minerals concerned can dissolve in one another, forming the so-called solid solutions. These are very common in refractories containing chrome, whilst the behaviour of chrome and chrome-magnesite bricks in steelplant furnaces is frequently associated with the solid solution of magnetite in the chrome grains.

*(c) X-ray standards*

Much of the vast amount of X-ray diffraction data now available has been condensed in the A.S.T.M. index. By using the cards supplied, substances can be identified in terms of the interplanar spacings corresponding to the diffraction lines on the powder photographs, together with their intensities; whilst as a preliminary to finding the right index card, reference may be made to a book containing a group numerical index. Where the possible nature of the compound is known use can be made of the alphabetical classification.

The patterns of the more common structural types within the simple crystal systems, particularly the cubic, can often be identified on sight. Use can therefore sometimes be made of tables of compounds classified according to their X-ray pattern or structure type and giving simply the lattice parameters—in the cubic system the length of the edge of a unit cube—in place of extensive tables of interplanar spacings. Frevel and others have made compilations of this kind for cubic, tetragonal and hexagonal systems, whilst the comprehensive classification of crystalline substances on the basis of unit cell dimensions has been published by the Geological Society of America.

Where X-ray techniques are regularly employed the research workers concerned normally build up their own library of standard patterns, which can then be compared directly with those of materials under investigation. Since the same minerals frequently recur the amount of labour required to identify an unknown substance may often thus be minimised.

(d) *High temperature cameras*

The study of constitution by X-ray methods is generally carried out at room temperature, but it is clear that additional information would be gained if the phases present at operating temperatures could be determined. A number of cameras capable of such work have been devised, whilst one of them—that described by Goldschmidt and Cunningham—is likely to become a relatively standard piece of equipment for work up to 1400°C. Those who wish to work at higher temperatures are confronted with much worse problems, but cameras have been described for work up to 1500°C. by Valkenburg and McMurdie, and for work up to 2200°C. by Johnson. The use of such equipment not only permits the identification of the phases present at temperature but also enables transformation points and thermal expansion characteristics to be determined. An early example of such work is described in the next chapter, where reference is made to the use by Jay of a high temperature camera, to measure the expansion characteristics of raw quartz.

(e) *Advantages and limitations*

X-ray examination of refractories sometimes provides the only quick and satisfactory method of determining constitution, a notable example being the identification of gamma dicalcium silicate and hydrated lime in stabilised dolomite bricks. A very small amount of material (about 1/100 g.) is sufficient for this purpose, and in some cases this may be all that is available. Thus the inclusions found in steel are often minute, but their nature can frequently be shown by X-ray examination. This may reveal that refractories are to blame, or alternatively that contamination has come from deoxidation processes. Thus the non-metallic inclusions in one sample of steel were found to consist of almost pure alumina, which was then traced to the aluminium added to the ladle rather than to the refractories through which the steel was cast.

On the other hand it must be admitted that the X-ray method suffers from the limitation that a small amount of an impurity may remain undetected because the line intensity is insufficient. The amount

involved varies greatly with the mineral, being as much as 5 to 10 per cent. with crystals having a poor pattern, but as low as 1 per cent. for a highly crystalline material like quartz. Furthermore this tendency of certain phases to stand out may give a misleading impression of the relative proportions present. A further limitation lies in the fact that the X-ray provides little information regarding any glassy constituents present, since these give virtually no pattern. This may seem unimportant since unused refractories are normally crystalline, but it should not be forgotten that slagged materials may contain large amounts of glass.

*(f) Geiger-Muller counter techniques*

Although the photographic method is the one generally employed considerable use has recently been made of Geiger-Muller counter spectrometers. With this type of apparatus a direct record is obtained of the intensity of the diffracted X-rays, the peaks in the curve corresponding to the lines in the powder photograph. The technique is particularly useful, where routine checks in constitution are made and quantitative results are desired. The apparatus is then used to scan a few of the principal lines, the relative heights of peaks being related to the proportions of the different constituents. The minimum amount of material that can be detected in a mixture is considerably lower with this technique than with the photographic method.

#### CRYSTAL SIZE

X-ray diffraction patterns can also be used for the estimation of crystal size, the "pinhole transmission" or "modified Laue" method being employed. With the usual procedure, unscreened (mixed white and characteristic) X-rays are passed through a thin slice of the material. A stationary single crystal gives under these conditions a Laue pattern, whilst a few crystals would give a set of such patterns superimposed on one another. As the number of crystals in the beam is increased diffraction spots lying on Debye-Scherrer rings also appear, whilst the Laue spots become more and more numerous and eventually merge into a more or less continuous background. There is therefore a gradual transition from a single crystal pattern to a complete Debye-Scherrer ring. Crystal size determinations can thus be carried out over a considerable range provided the necessary standards have been established. In the range where the Debye-Scherrer rings become spotty a quantitative estimate of crystal size is possible, the calculation starting from the number of spots in the ring (*see* figs. 49a, b and c, p. 91).

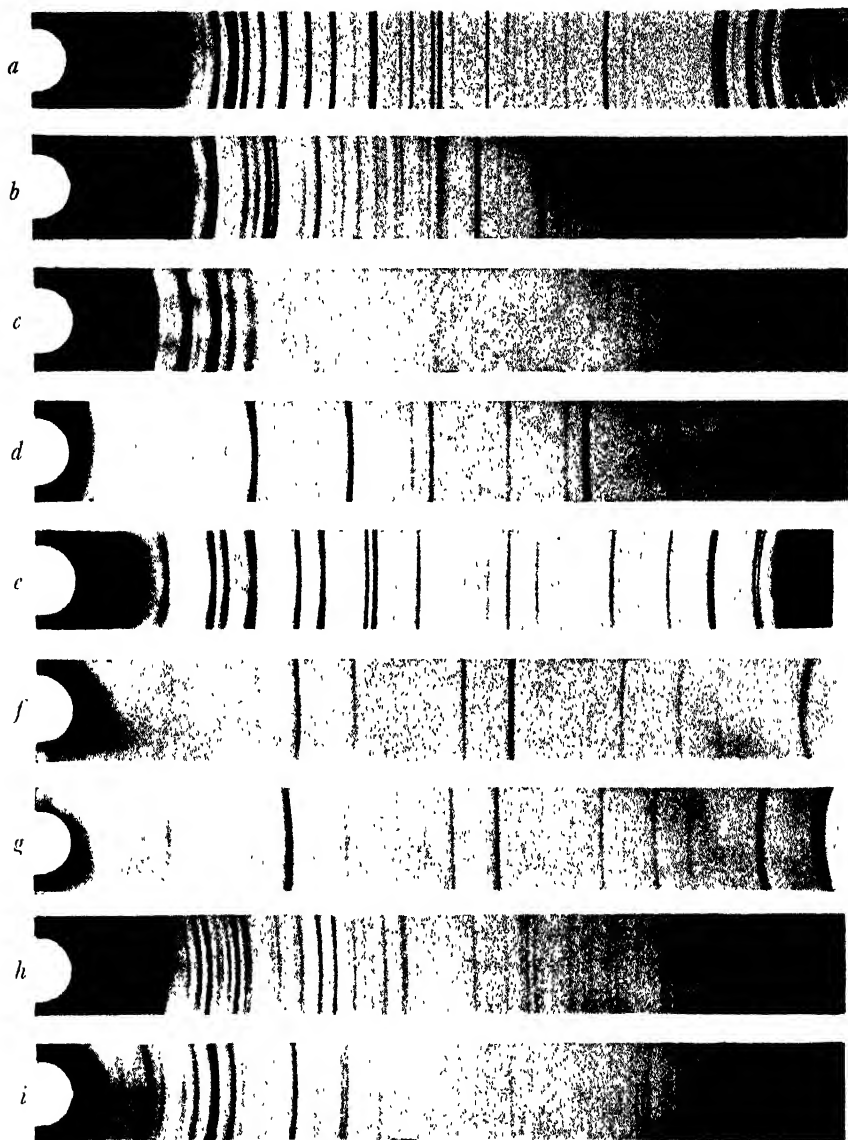


Fig. 12. Debye-Scherrer X-ray diffraction patterns of refractory materials. Note difference between patterns of  $\text{MgO} \cdot \text{Al}_2\text{O}_3$  and those of  $\text{NgO}$  and  $\text{Al}_2\text{O}_3$ , and similarity between  $\text{MgO} \cdot \text{Al}_2\text{O}_3$  and chromite.

- |                 |  |               |
|-----------------|--|---------------|
| a. Quartz       | d. Magnesia  | g. Chromite   |
| b. Cristobalite | e. Alumina   | h. Mullite    |
| c. Tridymite    | f. Spinel ( $\text{MgO} \cdot \text{Al}_2\text{O}_3$ ) | i. Forsterite |





An interesting application of crystal size measurement is given by chrome ore, where the pattern obtained is peculiarly characteristic of the place of origin. Thus ores from Greece show a coarsely crystalline structure, whilst those from certain other sources show an almost amorphous pattern. The method also provides a ready way of determining the crystallinity of dead-burned magnesite, which in certain cases, *e.g.* Grecian, may be difficult, if not impossible, under the microscope. X-ray examination of quartzites in general confirm microscopic examination, but may show in addition that certain of the larger quartz crystals are badly strained, or alternatively that these really consist of a large number of small crystals arranged at slightly different orientations. It is this observation that explains why some apparently coarsely crystalline quartzites show remarkably high conversion rates.

#### *Other X-ray techniques*

More recently increasing use has been made of radiographs both for detecting large faults in refractory shapes, *e.g.*, glass tank blocks, and for studying the distribution of phases within a thin section of the material. In this latter technique the refractory is photographed in contact with a piece of maximum resolution plate and the radiograph obtained magnified in an optical microscope so that the details can be studied on a scale similar to that employed in optical microscopy. Recent work on iron ores and sinters, which may be likened to the slagged face of a refractory brick, have shown that such microradiography frequently provides information not obtained with the ordinary microscope.

## DIFFERENTIAL THERMAL ANALYSIS

This specialised test, which is particularly useful in studying clay minerals, has made great progress in recent years and is now almost a standard part of ceramic laboratory equipment. The method depends on the observation of heat evolution or absorption, due to physical or chemical changes occurring in a material during heating. In clays such changes are particularly marked, Le Chatelier having made use of them many years ago to study the alumino-silicates. The work in Great Britain has been greatly furthered by the studies at the University of Leeds, where Houldsworth and Cobb developed their method, in which the test material was compared with the standard material rather than with the furnace itself. A good account of present-day methods was given by Grimshaw, Heaton and Roberts, in their 1945 paper on

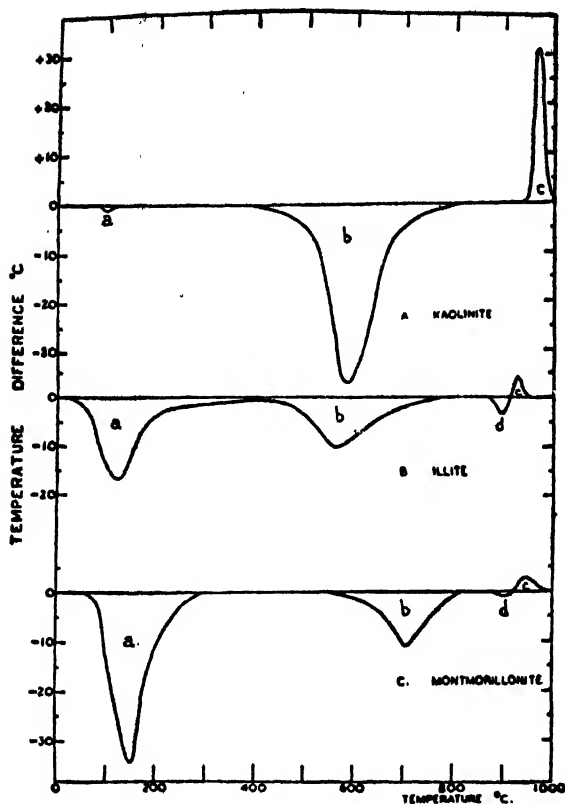


Fig. 13. Typical thermal analysis curves of clay minerals (after Grimshaw and Roberts).

"Thermal Analysis Methods". In their test a small sample, generally  $\frac{1}{2}$  to 1 g. in weight, is packed side by side with the reference material, usually recrystallised alumina, in two pockets in a metal block. More recently use has been made of sintered alumina blocks with a thin refractory wall between the two samples. Either base-metal or platinum type couples are used, according to the maximum temperature involved, whilst the furnace can be one of a number of types, always provided that it is capable of precise temperature control. The kind of results obtained with clay minerals is illustrated by fig. 13, which shows thermal analysis curves for kaolinite, illite, and montmorillonite. Other workers, *e.g.*, Howie and Lakin, have shown the method to be equally applicable to the study of changes in magnesite, dolomite and chrome products. They showed for example that the serpentine content of chrome ores

could be estimated from their thermal characteristics. More recently Grimshaw and Roberts have shown the possibility of using this tool in a still more quantitative manner, employing as a refinement the mixing with certain of the samples of a known proportion of inert material. They state that they can in this way determine the content of certain minerals to within  $\pm 1$  per cent.

Reference to papers given in the bibliography should suffice to show the potentialities of this tool and the speed with which its use is being extended. Other refinements will doubtless follow, *e.g.*, the atmosphere control suggested by Saunders and Giedroyd.

## SLAG RESISTANCE

The large number of factors controlling the rate at which a refractory is attacked by slag makes the reproduction of operating conditions in the laboratory extremely difficult if not impossible. This difficulty is reflected in the lack of any well known standard test method other than the German D.I.N. 1069, 1931. With steelplant refractories the problem is even more severe, in that the corrosive powers of iron oxide—the principal slagging material involved—varies greatly according to the state of oxidation, which in turn is influenced by furnace atmosphere or by contact with a molten bath. Slags obtained from furnaces for use in laboratory tests may not, therefore, remain typical of service conditions.

The ceramic technologist always approaches problems of slag attack, particularly with new refractories, by making use of any equilibrium data that are available. In particular he studies the melting points and primary phase relationships in the system formed by a join between the slag composition and the brick composition. Although this approach frequently provides useful information and may, for example, indicate that other things being equal a high alumina brick should behave better in contact with a given slag than a low alumina brick, it must never be forgotten that these are *equilibrium* diagrams and that they say nothing about *rates of reaction*.

## SLAG PILL TESTS

In the D.I.N. test referred to above, a hole 35 mm. deep and 44 mm. in diameter is drilled in the face of a test block, specified as having a horizontal surface of 80 mm.  $\times$  80 mm. and a height of 65 mm. Fifty grammes of finely ground slag are placed in the hole of the test piece and then given a specified heat treatment. After cooling it is sawn through and the extent of slag penetration and cut determined. Where, as in the rather similar test illustrated in fig 14, p. 31, the

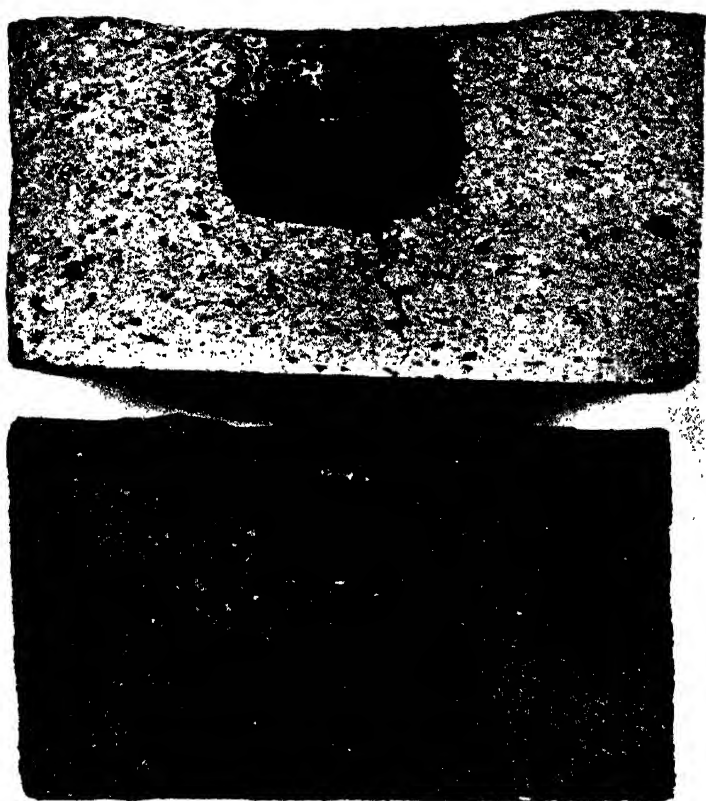
difference in cut with the materials is very marked, a positive answer is obtained, but frequently the main change is merely penetration and the seriousness or otherwise of this remains a matter of conjecture. In a still simpler but non-standard method of test, a pill of slag is placed on the flat surface of a brick and the reaction product examined as with the D.I.N. test. This method possesses the advantage that very little preparation of samples is necessary and that, therefore, a large number of different bricks can be tested in a relatively short time. It suffers, however, from a number of limitations, the worst of which is the tendency of fluid slags to run over the edge of the brick and attack the supporting bricks below rather than the test piece itself. A peculiar effect sometimes observed with certain slag-refractory combinations in these tests is the "drainage" from the slag pill of the more fluid constituents or reaction products, leaving behind a solid material.

#### REFRACTORINESS OF SLAG-BRICK MIXTURES

Where no equilibrium data are available some idea of the probable effect of a particular slag on a brick can be obtained by determining the slumping point of a series of cones or pills made up from brick-slag compositions, varying from 0-100 to 100-0 in 10 per cent. steps. Where the pill is of a type liable to attack the supporting brick it should be separated from it by a thin ( $1/100$  mm.) sheet of platinum foil. Tests of this type have, for example, been used to gain a rough impression of the influence of varying proportions of manganese and iron oxide on the melting point of various alumino-silicate compositions. The diagram given in fig. 15, p. 32, shows the effect of these oxides on a 40 to 60 alumina-silica composition, from which it can be seen that manganese oxide in quantities of up to 30 per cent. has a bigger effect in reducing the melting point of the alumino-silicate mix than does ferrous oxide. On the other hand a low melting point trough runs across the system parallel to the FeO-MnO base-line at a composition representing roughly 50 per cent. of flux addition. The melting point of this low temperature region falls below  $1200^{\circ}\text{C}$ . Information of this type frequently enables preliminary action to be taken and affords a useful—though by no means final—indication of the liquidus surface in the particular system concerned.

#### SLAG SPRAY TEST

A number of workers, *e.g.*, Hursh and Vickers, have employed tests in which finely ground slag is blown on to the surface of a row of bricks with a view to comparing their corrosion resistance. There is no doubt



*Fig. 14. Slag tests on fireclay (above) and plumbago (below) ladle bricks, made by heating slag in hole drilled in brick surface.*

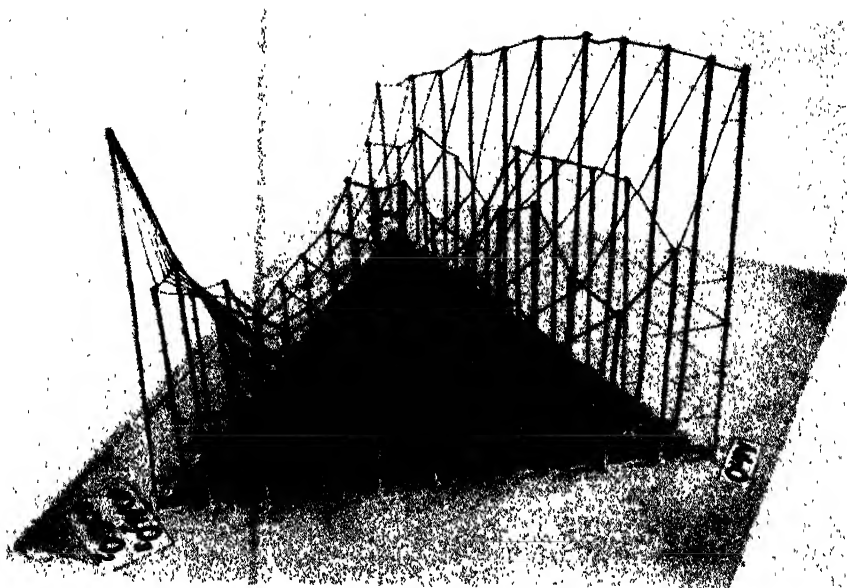


Fig. 15. Melting points in the system  $\text{FeO}-\text{MnO}-40-60 \text{ Al}_2\text{O}_3\cdot\text{SiO}_2$



Fig. 16. Induction furnace slag tests. Left to right: Fireclay, sillimanite, plumbago (used and unused) after similar periods of contact with molten bar iron and iron oxide slag.

that such tests simulate service conditions more closely than those described above, but the test procedure is rather lengthy and the results difficult to express in quantitative terms.

#### DIP ROD TEST

A great deal of useful information has been obtained, particularly on glass tank refractories, by the partial immersion of refractory rods in pots of molten glass, or the like. A similar test has been used recently, for example in France, Great Britain, and Italy, to assess the performance to be expected from different types of ladle bricks. (Further details of this are given in Chapter XIV.) In certain tests the rod is immersed in molten slag, whilst in others it is dipped through slag into molten metal in an induction furnace. The correlation obtained with service results is none too close, but even so the test is useful in that it can show up the effect of major changes, *e.g.*, in porosity, on the durability of a particular type of refractory.

#### INDUCTION FURNACE TEST

Where refractories are to be used for holding molten metal, *e.g.*, furnace hearth materials, a test in which molten metal, either with or without a slag coating, is maintained in contact with the particular refractory, clearly possesses a marked advantage. Such a test can most conveniently be done by means of a small induction furnace, the refractory being used either as a monolithic lining or in the form of a crucible. Fig. 16 opposite illustrates the kind of results obtained with such a test, indicating, for example, the superiority of plumbago compared with fireclay in resisting attack by molten metal, a result amply confirmed by large-scale trials.

The most that can be said of all the above slag tests is that they reveal trends or potentialities in materials that must be subsequently confirmed or disproved by service trials. For this reason many steelplant operators prefer to carry out their tests on actual furnaces. One of the positions frequently used is the end wall, and in particular the target area, on an open-hearth furnace which is bombarded by iron oxide laden gases at temperatures around 1700°C. A brick capable of standing up in this position for a longer period than the standard material is likely to give good service almost anywhere in an open-hearth furnace chamber, except perhaps in the roof, where arch stresses may be the cause of failure rather than slag attack. Tests on ladle bricks are



also frequently carried out in full size ladles, commencing with a few bricks, proceeding to panels and finally half or complete ladles.

With aluminosilicate refractories, the action of the slag is almost entirely one of solution, but with other refractories, *e.g.*, stabilised dolomite, the ultimate cause of failure may be quite the opposite, *i.e.*, the build-up of a slag layer to a point where cleaning of the surface becomes necessary. Similar considerations often apply to basic refractories in open hearth furnaces, where disruption, for example, of chrome-magnesite bricks, may precede any obvious solution. Examples of this type of attack are given in more detail in Chapter V.

## RUPTURE AND ELASTICITY TESTS

Although a rough picture can be obtained of both the cold and hot strength of refractories by the use of standard cold crushing and refractoriness-under-load tests, there are numerous occasions when more specialised information is desired. Use can then be made of alternative procedures such as those described below:

### (a) MODULUS OF RUPTURE

Failure of a brick in practice rarely takes the form of a cold crush, in general bending moments are involved and the brick fails by breaking in two rather than by crushing. The A.S.T.M. Standards on Refractory Materials contain an agreed procedure for this type of test, which possesses certain advantages over the cold crushing procedure, *e.g.*, it does not demand as powerful a testing machine, is more rapid—in that no special preparation of the bricks is required—and is in general a cleaner operation. The essential idea of the test is shown in fig. 17, from which it will be seen that the brick is supported on two bearing cylinders placed 7 in. apart and the load applied by a bearing cylinder of  $\frac{3}{8}$  in. radius located centrally on the top surface. The speed of the head on the testing machine should not exceed 0.05 in. per minute, or the rate of load application when using a hydraulic machine should be 4000 lb. per minute.

The modulus of rupture ( $R$ ) in lb. per sq. in. is reported as  $R = \frac{3wl}{2bd^2}$  where  $w$  is the total load in lb. at which the specimen failed,

$l$  is the distance between supports in inches,

$b$  is the width of the specimen in inches,

$d$  is the depth of the specimen in inches.



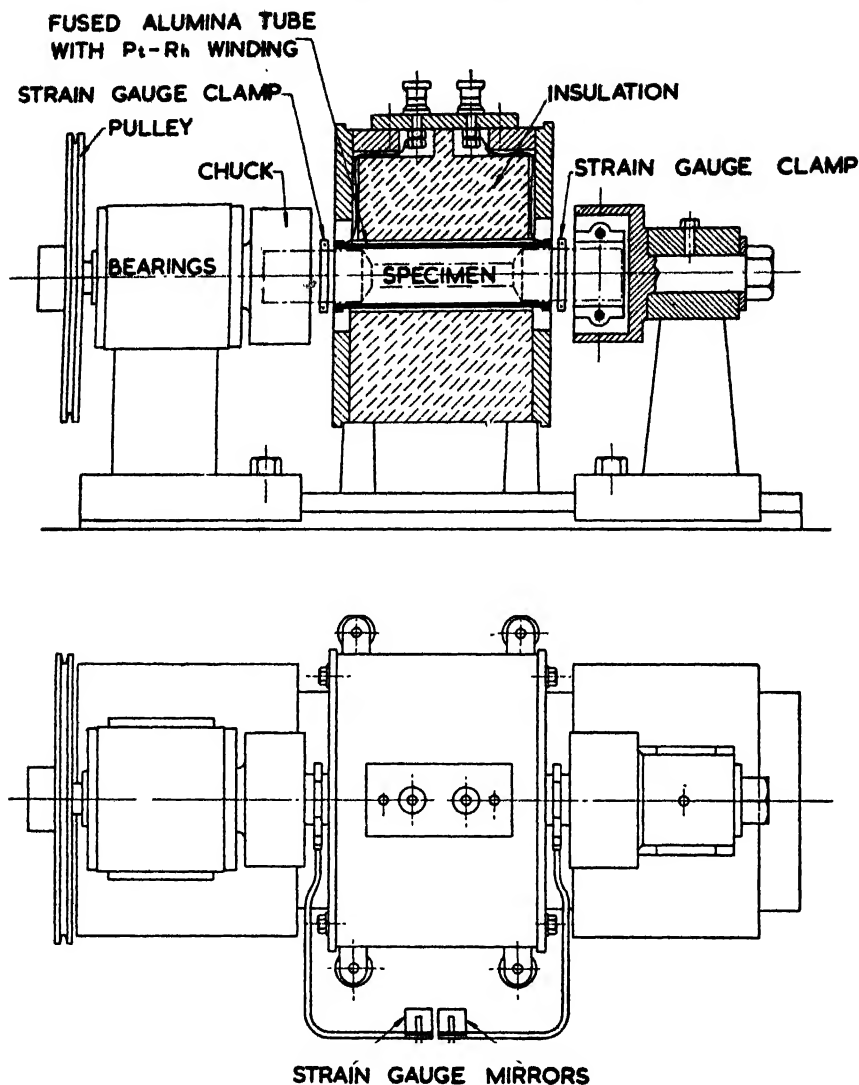


Fig. 18. Torsional elasticity apparatus (after A. L. Roberts).

Other types of creep test have been employed, *e.g.*, Mong, in his studies on a range of alumino-silicate refractories used both tensile and compressive loads applied for periods of up to 240 days. Recent tests in Great Britain have also determined long term creep using test pieces and procedures similar to those employed with heat resisting steels.

## (c) YOUNG'S MODULUS

Baab and Kraner described in 1948 their sonic method for determining Young's Modulus of Elasticity, which possesses the advantage of being non-destructive. This technique is really a refinement of the very old craftsman's trick of striking a brick with a hammer and deciding by the note emitted whether the brick was or was not hard fired. With magnesite bricks, for example, the confidence shown in this method has even extended to its inclusion in a specification.

The method described by Baab and Kraner, which is now in use in a number of laboratories, can be understood by reference to fig. 20,

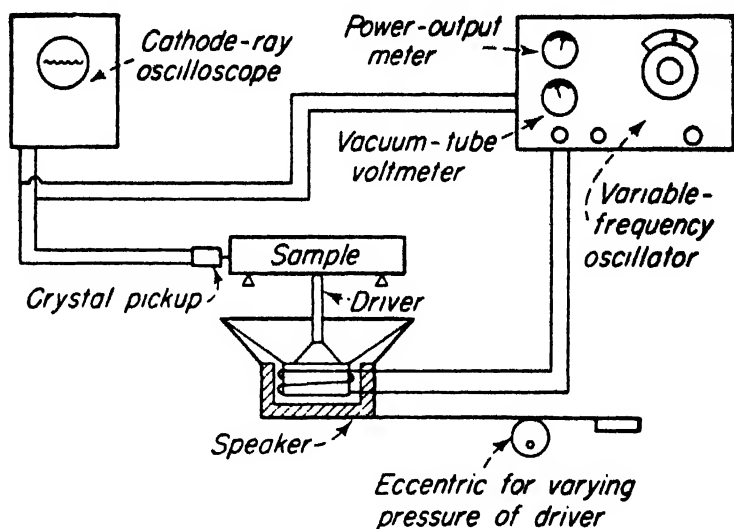


Fig. 20. Schematic diagram of sonic modulus of elasticity equipment (after Baab and Kraner).

which gives a diagrammatic sketch of the equipment. It will be seen that in this particular installation a moving coil loud speaker is employed to apply a signal to one face of a brick, whilst a crystal pick-up coupled to a cathode ray oscilloscope is used to record the response. If now the speaker is fed from a variable frequency oscillator it will be found that when the natural resonant frequency of the brick is attained the crystal pick-up shows a maximum response. The modulus of elasticity can then be calculated from Pickett's formula quoted:

$$\text{Modulus of elasticity (lb. per sq. in.)} = C \times W \times N^2$$

where  $C$  is a factor depending on the shape and size of the sample, the mode of vibration and Poisson's ratio,

W is the weight (lb.),

N is the resonant frequency in cycles per second.

Using this technique Kraner was able to show a close correlation between modulus of rupture as determined by the A.S.T.M. test and modulus of elasticity data obtained by the sonic method. He also showed that both types of modulus increased fairly steadily with firing temperature. The sonic method provides a non-destructive way of testing the degree of firing of batches within a consignment and provides a quantitative alternative to the original hammer blow and hoped for "Hell Klang" or clear ring.

## ABRASION RESISTANCE

Much of the wear in open-hearth furnaces loosely attributed to abrasion is almost certainly the result of slag attack, iron-rich droplets being deposited on the refractories by eddy diffusion or sudden changes in gas direction. There are however certain positions, for example, reheating furnace hearths, where abrasion resistance can play a more direct part. The advantage of carrying out abrasion tests however, is more often in the peculiarities revealed, *e.g.*, poor manufacturing control resulting in soft centres or loosely bonded grog. The kind of results obtained are well illustrated in the papers published by Baab and Kraner in 1948 and Mackenzie in 1951. The former made use of sandblasting equipment, 4 to 24 mesh silica sand being blown at an angle of  $45^\circ$  on to the 9 in.  $\times$  2½ in. face of a brick, at a velocity of approximately 150 ft. per second, whilst the face of the brick was moved slowly in a vertical 4½ in. stroke to spread the abrasion over a greater area. The rate of abrasion was expressed in terms of the loss in weight per hour. Mackenzie in his test made use of a proprietary machine employed for the peening of laminated springs. This apparatus although admirably suited for abrasion testing is unfortunately not one normally available to refractories technologists. In this method of test the bricks are placed on a conveyor belt which moves at a constant speed under an impinging jet of ½ in. chilled cast iron shot. Where the abrasion after one pass is insufficient to permit of the desired comparison, two or three passes may be employed.

More recently Cowling has developed an equipment similar to that employed by Kraner but rather simpler in design. With this apparatus, which is shown diagrammatically in fig. 21, the air blast which carries the abrasive is supplied by a 1 h.p. motor and the grit itself fed by gravity from a hopper into an annular chamber surrounding and communicating with the throat of a Venturi through which air passes.

As with the Baab and Kraner test impingement occurs at an angle of  $45^\circ$  but much smaller samples, having an area 3 in.  $\times$  2 in. and 1 in. thickness, are employed. Cowling found a high correlation between the abrasability indices with this machine and those obtained by Mackenzie on similar products. Incidentally to avoid any silicosis risk Cowling replaces the sand used by Baab and Kraner with angular fused alumina as manufactured for abrasives.

It is encouraging to note that both Baab and Kraner and Mackenzie found a marked relationship between the loss in their abrasion tests and the corresponding modulus of rupture: the former falling rapidly up to a rupture figure of 2000 lb. per sq. in. and then more gradually.

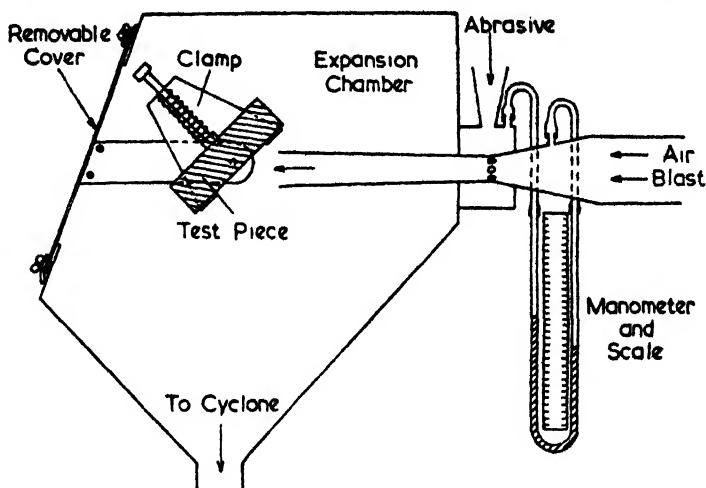


Fig. 21. Abrasive blast machine (after Aldred, Elliott and Cowling).

It would appear therefore that in the absence of abrasion test equipment, modulus of rupture tests afford a possible alternative. They would not, however, show up the peculiar variations in abrasion resistance within a brick, such as are illustrated in fig. 22, p. 42.

## THERMAL CONDUCTIVITY

The precise methods used for the determination of thermal conductivity at low or high temperatures depend greatly both on the materials being studied and the degree of accuracy required. For ordinary day-to-day tests on steelplant refractories or insulating materials, the methods described below have been found satisfactory and fair agreement exists between the results obtained by different procedures.

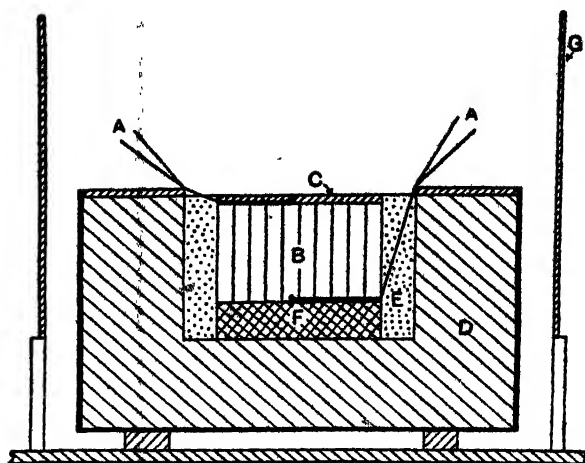


Fig. 23. Low temperature thermal conductivity apparatus—T. H. Blakeley and J. W. Cobb type. (A) thermocouples, (B) test brick, (C) brass plate with blackened top, (D) diatomite bricks, (E) diatomite powder, (F) heating element, (G) draught shield.

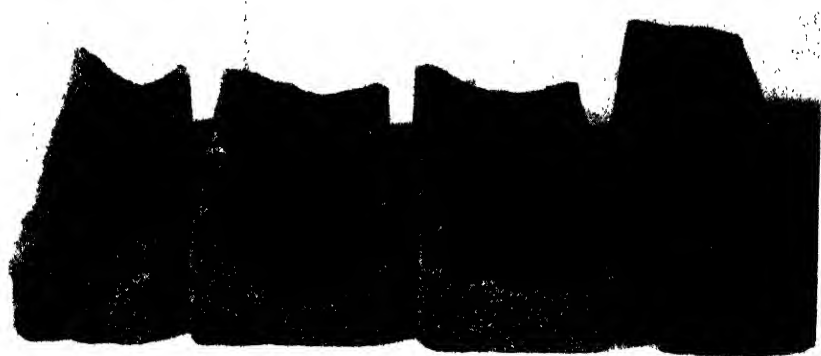
#### (a) LOW TEMPERATURE

The apparatus shown diagrammatically in fig. 23, which was used to obtain most of the conductivity data given in Chapter VII, is essentially that devised by Blakeley and Cobb and consists essentially of a flat electrical heater made by winding heat resisting wire on a 1 in. fireclay split, the test brick—normally a 9 in.  $\times$  4½ in.  $\times$  3 in. or 2½ in.—and a brass plate blackened on the top surface. These elements are surrounded by a box lined with diatomite bricks, the gap between the bricks and test piece being filled with diatomite powder. Temperatures are obtained by means of thermocouples and it is assumed that the heat passing through the brick escapes through the blackened surface of the plate and that at least in the central portion of the test piece the heat flow is parallel. Since the thickness of the brick and the temperature drop across it are known, the conductivity can then be readily calculated from the heat flow formula and tables showing heat losses from blackened horizontal surfaces in still air. Where greater accuracy is required use can be made of a modified apparatus, such as that developed by Clements and Vyse and shown diagrammatically in fig. 24. This makes use of peripheral heaters to reduce any side losses and replaces the blackened steel plate by standard water flow calorimetry.



*Fig. 19. Torsional elasticity apparatus (after A. L. Roberts).*





*Fig. 22. Fireclay bricks after Mackenzie abrasion tests.*

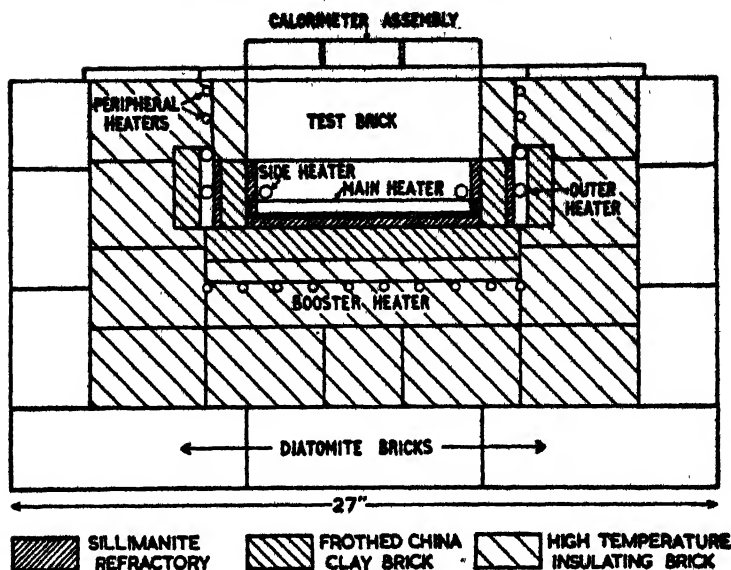


Fig. 24. Vertical section through high temperature conductivity thermal apparatus (after Clements and Vyse).

#### (b) HIGH TEMPERATURE

Although a great deal of research has been done in recent years on methods of determining the thermal conductivity of refractories and precise data are now available for a whole range of new materials, the apparatus designed by Patton and Norton, *see* fig. 25, is still the nearest to standard for refractories. Indeed it is virtually identical with that described in the A.S.T.M. designation C201-47. The test pieces in this method are heated from the top face by means of silicon carbide type resistor rods and rest on a complex water calorimeter and guard ring. The heat passing through the bricks is measured by the rise in temperature of the cooling water, whilst the temperature gradient is directly measured. The apparatus is stated to be capable of giving satisfactory results for materials with a conductivity of up to 15 B.Th.U./hr./sq. ft./°F./in. thickness. Details of the method employed and in particular of the precautions necessary if accurate results are to be obtained are set out in the above mentioned standard. In many of the recent methods, use is made of special shapes, *e.g.*, spheres and ellipsoids, which may well be suited to the study of new types of refractories, particularly where the latter are only available in small quantities, but are not so useful where the original samples are in the form of standard squares.

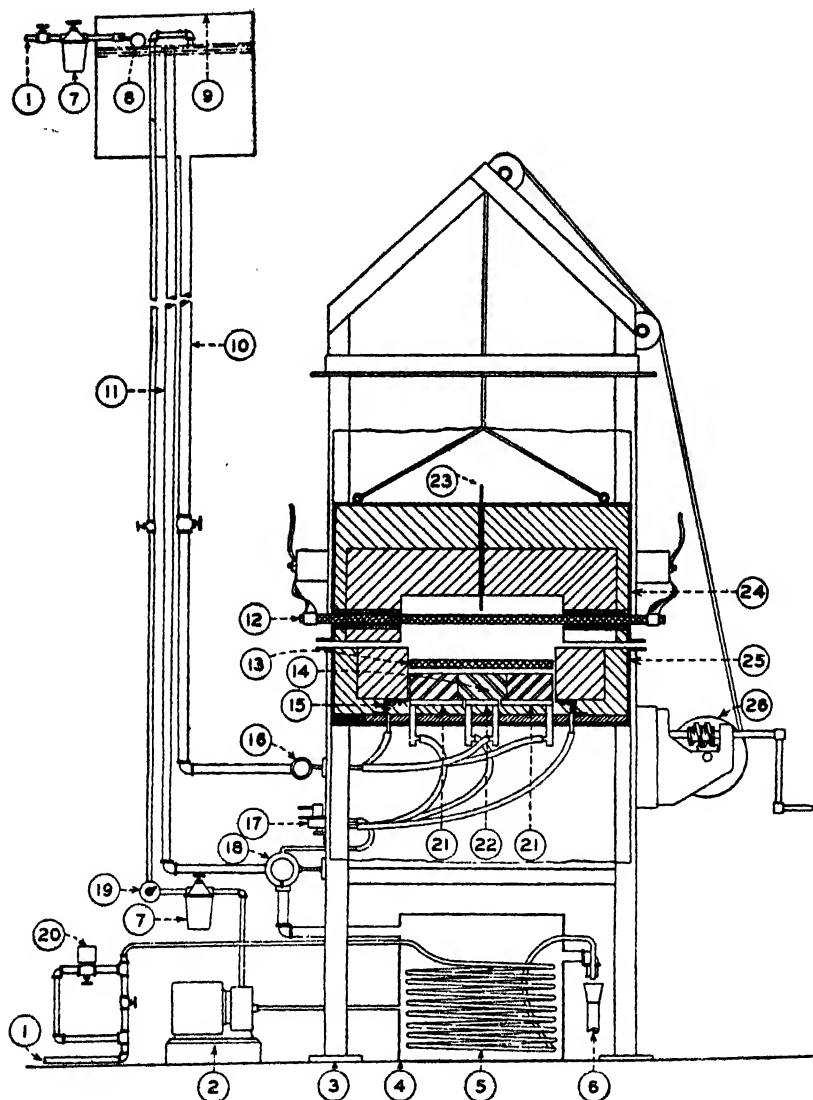


Fig. 25. High temperature thermal conductivity apparatus—T. C. Patton and C. L. Norton, Jr. (1) Connections to water supply, (2) water pump, (3) frame, (4) cooling tank, (5) cooling coil, (6) connection to drain, (7) water filter, (8) float valve, (9) constant-head water supply tank, (10) inlet pipe, (11) overflow pipe, (12) Globar heating elements, (13) silicon carbide slab, (14) test-specimens, (15) outer guard, (16) inlet manifold, (17) micro-regulating valves, (18) outlet manifold, (19) thermostat, (20) magnetic control valve, (21) guard calorimeters, (22) centre calorimeter, (23) control thermocouple, (24) heating section, (25) tester section, (26) winch for elevating heater section.

Those who wish to study such more specialised test methods are referred to the recent Symposium published in 1954 by the American Ceramic Society, in vol. 37, No. 2, Part II, of the Journal.

## SPECIFIC HEAT

The specific heat of refractories is rarely determined, mainly because for a given type of material it is relatively constant. If, however, data are required on new materials these can be obtained by the normal "Method of Mixtures". Where very precise results are required attention must be given to numerous details described, for example, by W. P. White in his classical work on the specific heat of silicates. For most purposes, however, and in particular for the calculation of heat storage in brickwork, the more elementary methods in which a sample is heated to a known temperature and then dropped in a water calorimeter surrounded by a water jacket is sufficiently accurate. Three main errors can arise during the transfer from the furnace to the calorimeter: *viz.*, loss of heat from the specimen before hitting the water, splashing of water, and loss of heat due to steam formation. The first of these can, as Clements suggests, be dealt with by enclosing the specimens in a platinum capsule which is first dropped empty to give a blank reading, whilst the splash can be minimised by the use of a guard around the calorimeter rim. Steam formation can be almost eliminated by allowing the sample to drop into a small metal receptacle floating in the calorimeter water with just its rim protruding. With this device no water touches the specimen and therefore no steam is produced until the surface of the water has closed over the submerging receptacle.

## MISCELLANEOUS

Numerous other tests are employed in the evaluation of refractories, *e.g.*, those developed for the testing of cements, concretes and castables. Certain of these, *e.g.*, those used to determine the hydration tendency of magnesia and the bursting expansion of chrome-magnesite bricks, are dealt with in the chapters on materials, whilst others, *e.g.*, those employed for testing induction furnace linings, are described in connection with the corresponding type of furnace.

## STATISTICAL TREATMENT OF TEST DATA

The extensive testing of refractories both by manufacturers and users has given rise to numerous theories regarding the effect of brick properties on service behaviour. Many of these are doubtless sound, but

others require careful checking in the light of the two main variables, *viz.*, conditions of manufacture and conditions of service. In the first group must be included variations in properties within a batch and between batches of bricks, and in the second variations in operating conditions within a furnace campaign, from one campaign to the next, and as between furnaces in the same or different works. The errors arising from testing are generally negligible compared with such uncontrolled fluctuations in brick quality and furnace operation. The practical man has always made some allowance for such variations when drawing conclusions from trials, but where these become serious recourse must be had to statistics if a quantitative answer is required, or an objective check on the validity of the conclusions is to be obtained.

The science of statistics is not concerned with the accumulation of data, so much as with its mathematical treatment according to the rules governing chance fluctuations. By making use of the laws of probability, the variability can be stated in a precise manner and differences both in properties and behaviour determined. Those who wish to apply statistics to their problems will do well to consult the standard works such as those mentioned in the bibliography. The examples given below are merely intended to arouse interest in this particularly fascinating tool. It should be stressed even before considering these elementary examples that no amount of statistical treatment will give valid results from inaccurate data, though it may enable a measure of useful information to be got from figures that might otherwise prove useless or even misleading.

#### NORMAL OR GAUSSIAN DISTRIBUTION

Students of vital statistics, *e.g.*, birth and death rates, have long been familiar with the fact that the grouping of such data as the number of men in various height-groups, often leads to a characteristic frequency curve known as the Normal or Gaussian distribution. Thus there tends to be in, say, 1000 men selected at random, a large number of the average height of the population, a few very short men, a few very tall men and intermediate numbers between the average and the extremes. If the heights are plotted horizontally and the number of men in each group, *e.g.*, 5 ft. 8 in. to 5 ft. 9 in., vertically, the result is a bell-shaped curve. The greater the number of men in the sample, the closer is the approach to a truly normal distribution.

As an example let us consider the bulk densities of a large number of samples of calcined dolomite. Table I gives the results obtained on deliveries from one dolomite plant to one steel plant in a six-monthly

period. In the ordinary test procedure the average value and, possibly, the range are taken as a measure of quality. It will be seen that much more can be obtained from the data if it is treated statistically.

The values are first divided into a limited number of bulk density groups, the number in each of which is indicated on the frequency distribution diagram (or histogram) by the height (or area) of the corresponding rectangle. Those chosen for this particular analysis are shown in fig. 26. They vary in steps of 0.1 g.p.ml. from 2.2 g.p.ml. to 2.9 g.p.ml. One individual result (2.08 g.p.ml.) has not been plotted but has

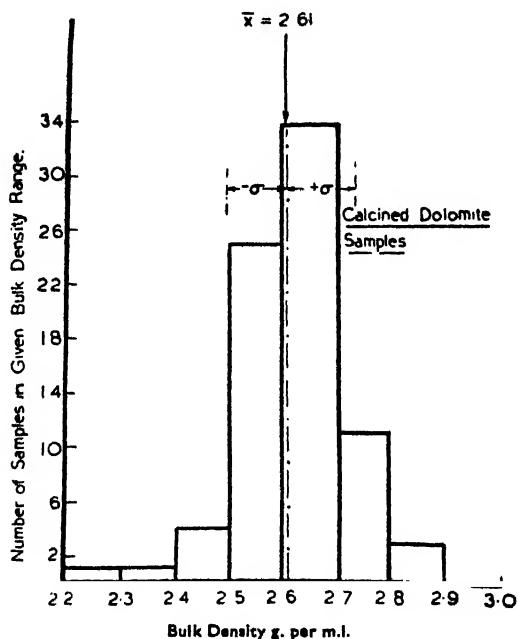


Fig. 26. Histogram showing number of calcined dolomite samples in each bulk density range.

been included in the calculations. It will be seen that the bulk densities, which tend to be high for hard-fired material and low for soft-fired material, do tend to form a bell-shaped figure. As is often (but not always) the case, the average value (or Mean) of 2.61 g.p.ml. falls in the "most frequent" group of 2.60 g.p.ml. to 2.70 g.p.ml. The more data available, the nearer the figure (or histogram as it is called) approaches a smooth curve. The distribution is not necessarily symmetrical about the mean. Thus if the specific gravities of hard-fired silica bricks are plotted, they give a skew distribution with the maximum point towards the low specific

TABLE I

BULK DENSITIES ( $\bar{X}$ ) OF CALCINED DOLOMITE DELIVERIES

$\bar{X}$	$(\bar{X}-\bar{X}) \times 10^3$	$(\bar{X}-\bar{X})^2 \times 10^4$	$\bar{X}$	$(\bar{X}-\bar{X}) \times 10^3$	$(\bar{X}-\bar{X})^2 \times 10^4$
2.75	14	196	2.63	2	4
2.73	12	144	2.60	— 1	1
2.65	4	16	2.74	13	169
2.63	2	4	2.61	0	0
2.52	— 9	81	2.62	1	1
2.67	6	36	2.65	4	16
2.29	— 32	1024	2.67	6	36
2.55	— 6	36	2.63	2	4
2.63	2	4	2.75	14	196
2.69	8	64	2.65	4	16
2.56	— 5	25	2.67	6	36
2.61	0	0	2.64	3	9
2.55	— 6	36	2.67	6	36
2.60	— 1	1	2.51	— 10	100
2.65	4	16	2.54	— 7	49
2.75	14	196	2.71	10	100
2.55	— 6	36	2.68	7	49
2.65	4	16	2.54	— 7	49
2.66	5	25	2.64	3	9
2.49	— 12	144	2.62	1	1
2.58	— 3	9	2.54	— 7	49
2.50	— 11	121	2.50	— 11	121
2.69	8	64	2.68	7	49
2.55	— 6	36	2.56	— 5	25
2.69	8	64	2.66	5	25
2.55	— 6	36	2.80	19	361
2.43	— 18	324	2.66	5	25
2.08	— 53	2809	2.82	21	441
2.73	12	144	2.77	16	256
2.57	— 4	16	2.72	11	121
2.35	— 26	676	2.61	0	0
2.64	3	9	2.59	— 2	4
2.54	— 7	49	2.79	18	324
2.51	— 10	100	2.48	— 13	169
2.68	7	49	2.59	— 2	4
2.52	— 9	81	2.79	18	324
2.58	— 3	9	2.69	8	64
2.40	— 21	441	2.58	— 3	9
2.51	— 10	100	2.57	— 4	16
2.61	0	0	2.81	20	400
102.89/40	—	7237/40	105.98/40	—	3668/40

TABLE I—*continued*:

Mean (average) value of  $X$ , i.e.,  $\bar{X} = \left[ \frac{102.89 + 105.98}{2 \times 40} \right] = 2.61 \text{ g.p.ml.}$

Range of  $X = 2.82 - 2.08 = 0.74 \text{ g.p.ml.}$

$$\begin{aligned} \text{Standard deviation} = \sigma &= \sqrt{\frac{\sum (X - \bar{X})^2}{N - 1}} \\ &= \sqrt{\frac{10905}{79 \times 10^4}} = 0.12 \text{ g.p.ml.} \end{aligned}$$

gravity side of the scale. This is because the specific gravity of silica bricks changes on firing from about 2.65 to a minimum of about 2.32. If they are all very well fired, they all approach 2.32. More often they tail off into the higher specific gravity ranges. With dolomite, on the other hand, the firing temperature, although exceedingly high, is never sufficient to reach the maximum bulk density or even to approach it at all closely. It is, however, well above the minimum value obtained with light-fired material, and hence the normal fluctuations of stone quality, and firing treatment, result in a normal distribution. Even if no more use is made of statistics than to plot a histogram, the data mean a great deal more than a mere list of figures with an average. Thus initial samples of silica bricks taken from a roof delivery were found to have specific gravities on the high side, but tests made on 20 bricks showed a similar histogram to that of a whole year's delivery of bricks indicating that the roof, as a whole, was as hard fired as usual.

Tests made on the cold crushing strength of various types of firebricks show similar fluctuations, due to variations in manufacturing procedure. Moreover, the better fired bricks show the greatest variations, probably because the crushing strength rises rapidly as the vitrification range is approached. With such materials, tests made on odd bricks are likely to be most misleading, and the B.S.I. suggestion of at least six determinations is a minimum precaution. The actual number of samples taken for a given test and the number of individuals within the sample will vary greatly with the job in hand, being smallest where the variability of the product is least. In extreme cases, where the variation is small compared with that which is of practical importance, testing can be discontinued, at least until some deterioration is suspected.

The use of histograms is also of great value in connection with size-variations. Thus, as a result of a large number of measurements made on the external diameter of guide tubes, it was shown that of three



manufacturers, one was making the desired size and keeping within close limits, a second was right on average, but subject to wide variation, whilst a third was very consistent, but consistently wrong. This relatively quick study revealed the best supplier (from the standpoint of shape), and also the supplier who could make equally well-sized guide tubes, provided his mould was suitably modified.

### STANDARD DEVIATION

Results showing approximately a normal distribution can be compared by measuring three quantities, *viz.*:

- (1) The average or arithmetic mean.
- (2) The standard deviation.
- (3) The skewness.

The first quantity is self-explanatory. The third is a measure of the extent to which the observations group themselves more on one side of the most frequent value than the other. The specific gravity of hard-fired silica bricks has already been mentioned as an example. Skewness can be expressed mathematically, but for the present it is sufficient to note its occurrence in the histograms.

The second quantity, the standard deviation ( $\sigma$ ), is most useful, because it describes the spread of the values on either side of the mean. It is defined for a large set of  $N$  numbers  $X_1, X_2 \dots X_N$  as the square root of the average of the squares of the deviations of the numbers from their average  $\bar{X}$ , *i.e.*:

$$\sigma = \sqrt{\frac{\sum (X - \bar{X})^2}{N - 1}}$$

The standard deviation from the mean of the 80 samples of dolomite mentioned above is worked out in Table I. It will be seen that the mean is 2.61 g.p.ml. and the standard deviation 0.12 g.p.ml. The direct value of this function lies in the fact that if the distribution is normal then the proportion of observations having a value:

- (i) less than  $\bar{X} - \sigma$  or greater than  $\bar{X} + \sigma$  is 15.9 per cent.
- (ii) „ „  $\bar{X} - 1.7\sigma$  „ „ „  $\bar{X} + 1.7\sigma$  „ 4.5 „
- (iii) „ „  $\bar{X} - 1.96\sigma$  „ „ „  $\bar{X} + 1.96\sigma$  2.5 „  
(often adopted for inner control limits)
- (iv) „ „  $\bar{X} - 3.09\sigma$  „ „ „  $\bar{X} + 3.09\sigma$  is 0.1 per cent.  
(often adopted for outer control limits)

Another way of expressing the distribution is that 31.73 per cent. of all the values fall outside the  $\bar{X} \pm \sigma$  limits 4.55 per cent outside the  $\bar{X} \pm 2\sigma$  limits and 0.27 per cent. outside the  $\bar{X} \pm 3\sigma$  limits. The 1.96 and 3.09 factors used above have the advantage that they correspond to the 1 in 40 and 1 in 1000 chances, but for most purposes, *e.g.*, the control chart on dolomite shown later, the practical results are much the same whether these limits or the  $2\sigma$  and  $3\sigma$  limits are used.

Two other values are often quoted, *viz.*:

#### RANGE

This is a measure of the total spread, *i.e.*, the difference between the highest and lowest values. For the above dolomite samples it comes to 0.74 g.p.ml.

#### COEFFICIENT OF VARIATION

This is merely the standard deviation expressed as a percentage of the mean. It is used in comparing the variability of different materials, and, unlike the standard deviation, is dimensionless. For the dolomite samples given in Table I, it is:

$$\frac{\sigma}{\bar{X}} \times 100 = \frac{0.12}{2.61} \times 100 = 4.6 \text{ per cent.}$$

#### SIGNIFICANCE TESTS

When data are to be interpreted, it is often found that they may be divided into several quite separate groups. For instance, they may have been collected at different times, under different conditions or before and after some process change, and the means or standard deviations will not be exactly the same for each group. The question then asked is: can the differences be reasonably attributed to chance variations, or should it be concluded that some real difference between the groups exists?

To answer this, a test of significance is applied. It is first assumed that there is no real difference and the probability of such results occurring if this were true is then calculated. If the probability is fairly high, the evidence available casts no doubt on the assumption, but if it is low, then it may be concluded that either a very improbable event has occurred or some real or significant difference exists.

The simplest significance test, known as a *t*-test, arises when the means of two groups of data are to be compared. The means  $\bar{X}_1$  and  $\bar{X}_2$

of the two groups of  $N_1$  and  $N_2$  results respectively are found, together with the standard deviation  $\sigma$

$$\sigma = \sqrt{\frac{\Sigma (X_1 - \bar{X}_1)^2 + \Sigma (X_2 - \bar{X}_2)^2}{N_1 + N_2 - 2}}$$

Then  $t = \frac{\bar{X}_1 - \bar{X}_2}{\sigma} \sqrt{\frac{N_1 \times N_2}{N_1 + N_2}}$  can be calculated, and the probability of

finding a value as high or higher if there is no real difference looked up in the appropriate statistical tables.

The data in Table II refer to open-hearth silica roof lives for two brickmakers measured in casts and corrected for the percentage

TABLE II  
OPEN-HEARTH UNIT ROOF LIFE IN CASTS

	<i>Brickmaker 1</i>	<i>Brickmaker 2</i>	
	<i>Oil-fired furnace</i>	<i>Oil-fired furnace</i>	<i>Pitch-cresote fired furnace</i>
	185	194	123
	223	251	84
	201	268	132
	209	193	
	215	245	
	181	160	
	264	231	
	196	158	
	214	226	
		171	
		163	
		242	
		181	
		140	
		193	
Number of results ..	9	15	3
Sum .. .. .	1888	3016	339
Mean .. .. .	209.8	201.1	113.0

repairs. These data were collected on similar oil-fired furnaces in one melting shop, with the exception of three results for maker 2 which were from a pitch-cresote-fired furnace. It is required to determine

firstly whether the means for the two brickmakers are significantly different and secondly whether there is a real difference between oil and pitch-cresote-fired furnaces for maker 2.

For maker 1 *versus* maker 2 on oil-fired furnaces,

$$\sigma = 35.1 \text{ and } t = \frac{8.7}{35.1} \sqrt{\frac{9 \times 15}{9 + 15}} = 0.59 \text{ which only reaches the } 55$$

per cent. probability level. This means that if, say, 24 results for one brickmaker were split into two groups of 9 and 15 results many times, 55 per cent. of the pairs of groups would show a difference between their means as high or higher than the value of 8.7 just by chance and the result is, therefore, not significant.

For maker 2, oil *versus* pitch-cresote,

$$\sigma = 38.4 \text{ and } t = \frac{88.1}{38.4} \sqrt{\frac{15 \times 3}{15 + 3}} = 3.62, \text{ which is significant at } 0.25$$

per cent. Here there is only 1 chance in 400 that such a large difference in the means could have arisen by chance and the evidence, therefore, strongly suggests that the use of pitch-cresote has had a detrimental effect upon roof life compared with oil firing, a conclusion supported by long-term differences in operating costs for oil and pitch-cresote-fired furnaces.

#### QUALITY CONTROL CHARTS

For the layman, the control chart on which new data are plotted affords the simplest means of setting-up quality control and deciding whether action is or is not required. Essentially, quality control charts are a means of comparing the original distribution of values on a material with the distribution at any subsequent period, *i.e.*, the variation in mean value and dispersion. This can be done by drawing a series of histograms one above the other, but interpretation of individual results is more clear-cut if a quality control chart is set up. In such a chart the ordinates represent the quantity measured, *e.g.*, in fig. 27 the bulk density of dolomite, whilst the abscissæ represent the sequence of tests, one space being given for each of the results irrespective of whether they were carried out at equal intervals of time.

The central horizontal line represents the mean, as determined by the initial study; in this case of the 80 samples of dolomite taken during the six-monthly period. The values of this mean plus and minus 2 and 3 times the standard deviation are drawn as inner and outer limits. If now it is desired to know whether the dolomite being received in a later period, is better or worse than in the previous period, the

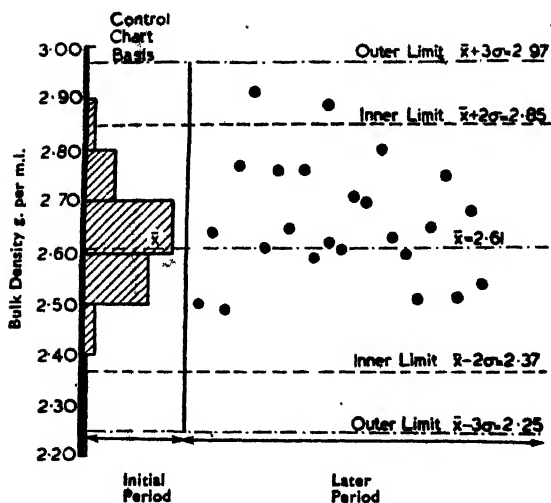


Fig. 27. Quality control chart for bulk density of calcined dolomite. Histogram plotted on ordinate shows original distribution. Dots show values obtained in subsequent period.

individual results are plotted in the manner shown. Even a glance at the chart suggests that the dolomite quality has improved, the bulk of the results being above the earlier mean, and no result below the  $\bar{X} - 2\sigma$  level. The probability of results falling outside these limits if the new samples came from the same "population" as the original samples used to set up the chart has already been shown to be quite low. Hence, if values occur repeatedly either above or below these limits, it can be assumed, with only a limited risk of error, that the material is better or worse than in the earlier period. Fig. 28 shows a quality control chart for alumina in silica bricks the time scale of which refers to periods of 2 months before and after introducing a washing technique. It shows not only a substantial drop in alumina (from 1.06 per cent to 0.82 per cent.) but also a more consistent product, the standard deviation falling from 0.14 to 0.07. The maintenance of such charts performs two functions not performed by the usual list of figures:

- (1) It avoids complaints, based on odd results, which may arise purely from inadequate sampling.
- (2) It often shows trends before they become serious, and enables appropriate action to be taken.

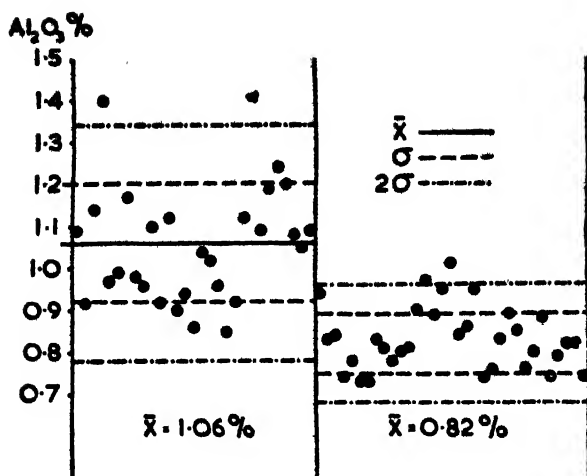


Fig. 28. Quality control chart for alumina content of silica bricks. Note sudden changes when rock washing introduced (after Lynam, Nicholson and Young).

#### CORRELATION COEFFICIENT

No abuse of scientific method is more common than the drawing of a straight line or a smooth curve through a set of points that do not warrant such treatment. True subsequent research often shows that the desired relation does in fact exist, but where the data is inadequate or the relationship very approximate, it is better to plot the points but not draw a curve.

Refractories is a good example of a subject that involves a large number of variables. The life of a silica roof brick may be influenced by its bulk density, its alumina content, the average working temperature, the quality of the steel being made, the number of hours above a critical temperature and probably at least 100 other variables. It should not, therefore, be expected that a simple relationship would be found between roof life and any individual factor. Thus it may happen that all the dense roofs are put on furnaces making low carbon steel. If so a straight plot of bulk density against roof life may suggest that porous bricks give a better roof life, whereas in fact the porous bricks have merely been subjected to easier working conditions.

It is often said that there appears to be some "correlation" between two factors. The degree of correlation can be expressed mathematically though it should be noted that, here again, we are completely dependent on the accuracy of the data, and even if we find a high degree of

correlation, we cannot always say which is cause and which effect or whether both are controlled by some third factor.

Let us, for example, assume that we have 2 lists of quantities, *e.g.*, the thermal conductivities of diatomite bricks and their bulk densities. Other factors, such as chemical analysis, will doubtless affect thermal conductivity, but a dot diagram (*see* fig. 29) does suggest a positive relationship. A table is then prepared of the type shown in Table III

TABLE III

CALCULATION OF CORRELATION COEFFICIENT BETWEEN BULK DENSITY AND THERMAL CONDUCTIVITY OF TWENTY DIATOMITE BRICKS

Bulk density g.p.ml.	$X - \bar{X}$	$(X - \bar{X})^2 \times 10^4$	Thermal conductivity— B.Th.U. at 400°C. mean	$Y - \bar{Y}$	$(Y - \bar{Y})^2 \times 10^4$	$(X - \bar{X}) \times (Y - \bar{Y}) \times 10^4$
$\bar{X}$			$\bar{Y}$			
0.81	+0.06	36	1.30	-0.03	9	- 18
1.24	+0.49	2401	3.16	+1.83	33489	+8967
0.52	-0.23	529	0.59	-0.74	5476	+1702
1.09	+0.34	1156	1.74	+0.41	1681	+1394
0.73	-0.02	4	1.00	-0.33	1089	+ 66
0.51	-0.24	576	0.70	-0.63	3969	+1512
0.44	-0.31	961	0.87	-0.46	2116	+1426
0.54	-0.21	441	0.98	-0.35	1225	+ 735
0.55	-0.20	400	0.84	-0.49	2401	+ 980
0.85	+0.10	100	1.55	+0.22	484	+ 220
0.53	-0.22	484	0.91	-0.42	1764	+ 924
0.64	-0.11	121	1.18	-0.15	225	+ 165
0.74	-0.01	1	1.22	-0.11	121	+ 11
0.76	+0.01	1	1.12	-0.21	441	- 21
0.92	+0.17	289	2.10	+0.77	5929	+1309
0.91	+0.16	256	1.66	+0.33	1089	+ 528
0.77	+0.02	4	1.23	-0.10	100	- 20
0.80	+0.05	25	2.00	+0.67	4489	+ 335
0.86	+0.11	121	1.24	-0.09	81	- 99
0.77	+0.02	4	1.29	-0.04	16	- 8
$\bar{X}=0.75$	—	$\Sigma(X - \bar{X})^2 \times 10^4 = 7910$	$\bar{Y}=1.33$	—	$\Sigma(Y - \bar{Y})^2 \times 10^4 = 66194$	$\Sigma(X - \bar{X})(Y - \bar{Y}) \times 10^4 = 20440$

$$r = \frac{\Sigma(X - \bar{X})(Y - \bar{Y})}{\sqrt{\Sigma(X - \bar{X})^2 \Sigma(Y - \bar{Y})^2}} = \frac{20440 \times 10^{-4}}{\sqrt{(7910 \times 10^{-4})(66194 \times 10^{-4})}} = 0.89$$

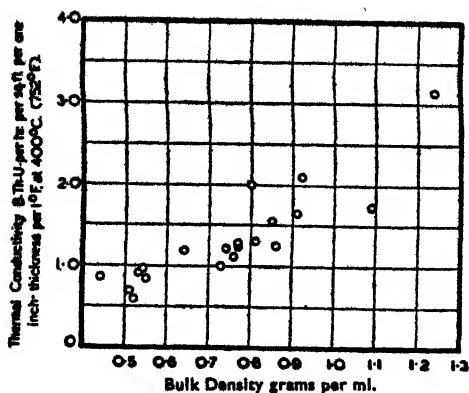


Fig. 29. Correlation (coefficient = 0.89) between thermal conductivity and bulk density of diatomite bricks.

where  $X$  represents the bulk density in g.p.ml. and  $Y$  the thermal conductivity at 400°C. mean temperature in B.Th.U. The values of  $(X - \bar{X})$  and  $(Y - \bar{Y})$  are then calculated and their squares. Also the values of  $(X - \bar{X})(Y - \bar{Y})$ .

The correlation coefficient for the results is then given by  $r$  where

$$r = \frac{\sum (X - \bar{X})(Y - \bar{Y})}{\sqrt{\sum (X - \bar{X})^2 \sum (Y - \bar{Y})^2}}$$

This is found to be 0.89 for the diatomite bricks, which indicates a close correlation. Had it been much lower, say 0.1, it would have suggested that bulk density had relatively little influence on conductivity. Had it been -0.89, it would have implied a high degree of correlation but a negative one, *i.e.*, when the bulk density rose the thermal conductivity fell. Perfect positive or negative correlation are indicated by +1.0 and -1.0 respectively. The zero value indicates no apparent relationship. Unfortunately, the precise significance to be attached to the coefficient depends on the number of values involved, and cannot be stated in simple terms. Those who wish to pursue the matter further are advised to consult the recognised text-books listed in the bibliography.

The above are relatively simple examples of the application of statistical methods. Far more complex problems involving perhaps a dozen factors can be studied, and the relative importance of these factors determined. The mathematics naturally became more complicated and sometimes wearisome, but since no other approach is available when quantitative answers are required, those who desire to tackle such problems must adopt this technique. For those to whom this spells discouragement it can be stated that the histogram stage, which anyone can reach, often provides 90 per cent. of the answer.



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## Chapter II

# SILICA AND SEMI-SILICA

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**A**LTHOUGH SILICA CONTINUES to meet severe competition from basic refractories of far higher melting point, it still maintains its position as the No. 1 steelplant refractory, due to the fortunate possession of a number of peculiarly desirable properties, in particular:

- (1) The ability to carry a substantial load (50 lb. per sq. in.) to within a few degrees of its cone fusion point, *viz.*, 1710° to 1730°C.
- (2) A high resistance to attack by the principal steel furnace fluxes, *viz.*, iron oxide and lime.
- (3) Freedom from shrinkage at temperatures up to the melting point.
- (4) A high thermal shock resistance in the temperature range 600° to 1700°C.

Its only serious limitation apart from its melting point is its sensitivity to thermal shock below 600°C., or below 300°C. in the case of a well-fired brick. This limitation is normally met by careful control in the heating up of silica structures, but does reduce its usefulness for certain purposes, *e.g.*, arc furnace roofs and door jambs, where thermal shock often leads to premature failure. There are also other positions, *e.g.*, reheating furnace fireboxes, where silica would probably be employed were it not for this unfortunate property. The potentiality for development is still far greater with basic refractories, but that silica itself has by no means reached its ultimate value has been dramatically shown by the big improvements recently obtained with bricks of lower alumina content and higher bulk density.

The semi-silica (or sand-clay) type brick also continues to hold a special place in the steelplant and partly for reasons that are still somewhat obscure. Its success would appear to be due in no small measure to the fact that, not only after firing but also frequently after use, it retains its original mixed quartz—fireclay structure rather than the relatively low melting point mixture of mullite and liquid predictable for equilibrium from the alumina—silica phase diagram.

SILICA—THE COMPOUND  $\text{SiO}_2$ 

It was recognised as early as 1900 that silica existed in three principal forms, *viz.*, quartz, cristobalite and tridymite. Much confusion has existed, and indeed still exists, regarding the precise relationship between these minerals and their different allotropic modifications, and such major issues as to, whether the ideal silica brick, say, for an open-hearth furnace roof, should consist of cristobalite, tridymite, or some mixture of these with quartz, remain unsettled.

It is particularly fortunate that a material possessing such remarkable properties should be so readily available. According to estimates quoted by F. W. Clarke silica constitutes no less than 59 per cent. of the outermost layer of the earth, though much of this is present in combination with other oxides giving materials that are far from refractory. It is known in its purest form as quartz, a name that to most people conjures up rock crystal of the type illustrated in fig. 30. p. 71. Such material, apart from its scarcity, is difficult to employ for the production of refractories, due to the fact that it requires a high temperature to give any great degree of conversion to the more permanent forms, *viz.*, cristobalite and tridymite. The rocks used in the manufacture of silica bricks also consist of quartz, but the crystals are far smaller and are usually cemented together by finely divided silica to form aggregates known as quartzites. X-ray examination by the Debye-Scherrer method shows that the grains in such quartzites have a lattice structure identical with that of sand or rock crystal. Even the gem stone amethyst possesses the same structure, though showing more perfect crystallinity and a distinctive purple colour due to traces of impurity. The external hexagonal structure shown by the massive quartz crystals in fig. 30, has been shown by X-ray examination to be due to the repetition on a large scale of an inner arrangement of atoms. Such an arrangement is shown diagrammatically in fig. 31*b*. It will be seen that the actual arrangement of the atoms is somewhat complex, but that when such a crystal is heated up to the point ( $575^\circ\text{C}.$ ) at which quartz does a sudden expansion, the atomic arrangement changes to simple hexagonal, as shown in fig. 31*a*. The comparison of the two pictures shows that there has been no disruption of the lattice, but merely a straightening of the linkages. It is this change at approx.  $575^\circ\text{C}.$  which is observed in the thermal expansion curves both of unfired silica material and of fired bricks containing raw quartz.

A. H. Jay, using a hot X-ray camera, was able to show (fig. 32) that the expansion in the quartz crystals was not the same for each crystal axis, as it is for example with cubic crystals such as magnesia. This observation offers a rational explanation as to why inversion

should occur when a crystal heats up, since relative displacement of atoms might well lead to a state in which the forces acting on them would no longer be in equilibrium and sudden reorientation might, therefore, occur. If heating continues to a temperature of  $870^{\circ}\text{C}$ . or over, it is found that the quartz pattern slowly disappears and another pattern, that of cristobalite, begins to take its place. X-ray examination shows that the new mineral has at high temperatures a cubic form, as

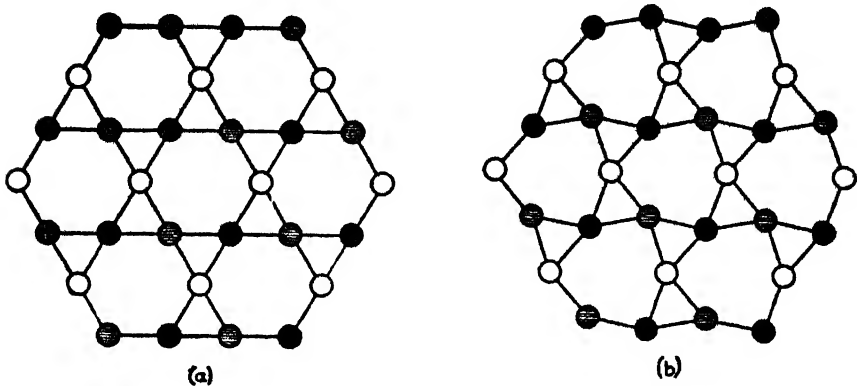


Fig. 31. Relation between (a)  $\beta$ -quartz and (b)  $\alpha$ -quartz. Silicon atoms only are shown (W. L. Bragg—*Atomic Structure of Minerals*).

shown in fig. 33, which also changes to a slightly different low temperature form on cooling. It should be emphasised that there has been no loss of either silicon or oxygen atoms in this conversion, but merely a re-orientation. This form of silica is particularly important to the steelplant operator, since it is the principal constituent of the working face (grey zone) of an open-hearth furnace roof. The conversion of quartz to cristobalite, or the third form—tridymite—can, as Dodd points out, only occur by the breaking of Si—O—Si linkages followed by rearrangement of the  $\text{SiO}_4$  tetrahedra. It is for this reason that the conversion is much slower than the changes observed within each particular form, such as the alpha-beta quartz change, which require only small alterations in bond direction and length. It has long been known that this conversion of quartz to cristobalite and tridymite, which is an essential part of silica brickmaking, can be considerably accelerated by the presence of mineralisers. A great deal of work for example was carried out by Hugill and Rees on this subject, the procedure being to make various additions and then compare the properties of bricks made both with and without them. If the experimental

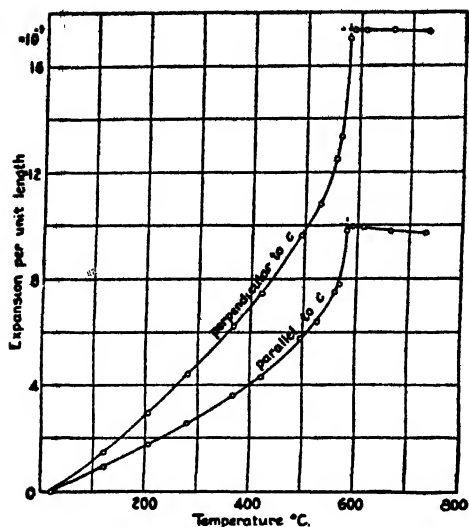


Fig. 32. Thermal expansion of quartz parallel and perpendicular to the  $c$  axis. Note the effect of the  $\alpha$ - $\beta$  transition at  $575^{\circ}\text{C}$ . (after A. H. Jay).

arrangements are such as to permit of continuous measurement of expansion due to conversion, then the effect of different mineralisers can be observed quite dramatically. Thus, Chesters and Parmelee showed that the addition of a mixture of 1.5 per cent.  $\text{Na}_2\text{O}$  and 1.5

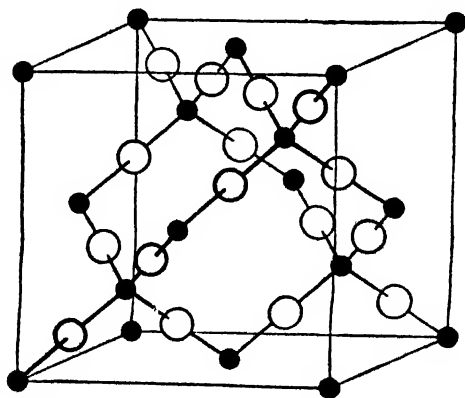
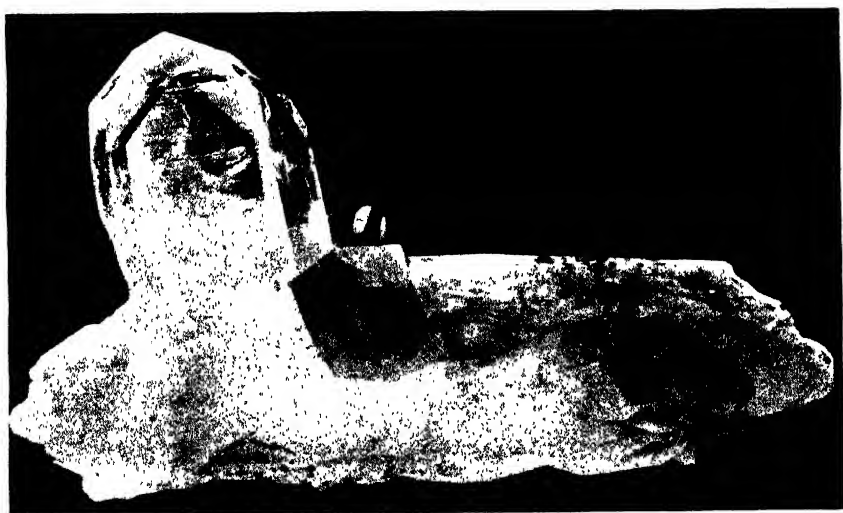


Fig. 33. The structure of  $\beta$ -cristobalite according to Wyckoff (W. L. Bragg—Atomic Structure of Minerals).



*Fig. 30. Massive quartz crystals approximately one-eighth actual size  
(F. A. Bannister, British Museum).*



*Fig. 34. Twinned crystal of tridymite in  
hard-fired silica brick. Crossed nicols  
×650.*

per cent.  $\text{Fe}_2\text{O}_3$ , a mineraliser mixture found by other workers to be particularly effective, produced as much conversion of quartz in 2 hours at  $1400^\circ\text{C}$ . as occurred in the absence of a mineraliser in 2 hours at  $1550^\circ\text{C}$ . The mechanism by which cristobalite and tridymite are formed in a silica brick and which was discussed very fully by A. J. Dale as far back as 1927, will be dealt with in a later section on the firing of silica bricks.

A study of the literature shows that many different temperatures are quoted for the inversion of cristobalite from the low to the high temperature stable form, and further that the precise temperature depends on the previous thermal history of the sample. Fenner, who worked out the phase relationships for various forms of silica as early as 1913, even went as far as to suggest that "cristobalite consists not of one but of two different molecular species in the same crystal". Subsequent workers have shown the presence in a given sample of more than one inversion in the cristobalite range, whilst A. L. Roberts and co-workers have recently demonstrated by differential thermal analysis the presence of conversion peaks at  $229^\circ$  and  $253^\circ\text{C}$ . in a single sample of cristobalite. The position is further complicated by the repeated observation that under certain conditions the high temperature form of cristobalite does not invert on cooling. It is possible that some of these effects would not be observed in absolutely pure material, but they are certainly part of the picture facing the ordinary user of steelplant refractories.

A further complication, is that beta cristobalite if held for an appreciable time in the temperature range  $870^\circ$  to  $1470^\circ\text{C}$ . tends to change to tridymite, the mineral shown as arrow head twin crystals in fig. 34, p. 72. Many workers in the field have observed that although this form is most readily recognised as tridymite, the latter also exists in a lath-shaped form. Much research has been done on the influence of mineralisers and temperature on tridymite formation, in the hope of producing a 100 per cent. tridymite brick that would not be subject to the sudden and more substantial cristobalite change.

The precise specific gravity of the pure forms is still a matter of discussion, but for practical purposes, the following specific gravities may be assumed:

Quartz	..	..	2.65
Cristobalite	..	..	2.33
Tridymite	..	..	2.27
Quartz glass	..	..	2.21

The fact that the specific gravity of quartz is markedly greater than that of tridymite and cristobalite affords a useful means of estimating



at least approximately the amount of residual quartz in a silica brick. Between the publication of the first and second editions of this book no less than 7 new phases of silica have been discovered, bringing the 1955 total to 15. Of these one new form, named Coesite by Sosman because of its discovery by Leonard Coes, is 14 per cent. denser than quartz and unlike other forms of silica possesses the astonishing property of being almost completely insoluble in aqueous hydrofluoric acid. At present, however, it is only of academic importance to the steelplant, since it was produced under pressures of over 35 kilobars. Sosman's new classification of the phases of silica at atmospheric pressure is reproduced as fig. 35. It will be seen that this contains another newcomer, *viz.*,

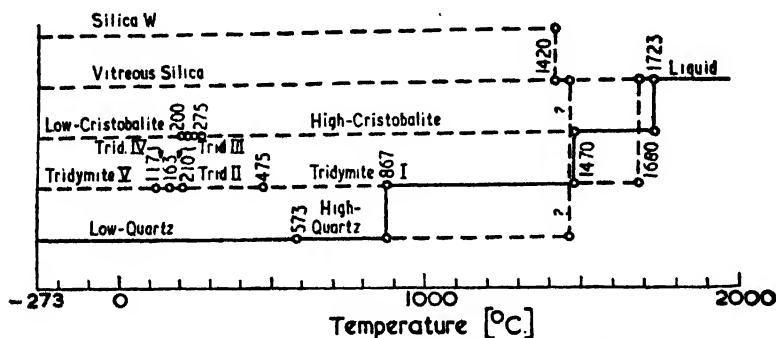


Fig. 35. Classification of the phases of silica at 1-atmosphere pressure, as revised by Sosman in 1955. Solid lines represent stable state, dashed lines represent meta-stable state.

Silica W, which has been prepared by the condensation of gaseous silicon monoxide. It takes the form of micro-crystalline fibres having the usual tetrahedral arrangement of 4 oxygen atoms about each silicon. It differs from the other forms of silica, however, in that the tetrahedron are joined at the edges instead of at the corners.

In passing it should be mentioned that comparatively recent research, *e.g.*, the paper by Beck, has demonstrated the extraordinary similarity in behaviour of aluminium orthophosphate ( $\text{AlPO}_4$ ) and silica. This mineral has been shown to be capable of existence in a "tridymite" and a "cristobalite" form in addition to the low temperature berlinite, though the conversions from one form to another are more rapid than with silica and the temperatures of conversion different. No large scale application appears to have been made of this knowledge as yet but it is more than possible that it may be of importance in the future in connection with the manufacture or use of silica bricks.

In both Great Britain and the United States, the general view of the steelman is that the brickmaker should fire silica bricks sufficiently to convert substantially all the raw quartz to either cristobalite or tridymite, so that he will not be faced in service with a serious after-expansion. If such growth in service were of a reasonable order, say,  $\frac{1}{2}$  per cent., and consistent for all bricks, it could be allowed for, but unfortunately once permission is given for medium or soft fired bricks to be supplied there exists the risk of quantities of very soft material being delivered, with consequent localised excessive expansions and shelling. In Germany, on the other hand, as has been emphasised again recently by Konopicky, the steelman seems to prefer a medium fired silica brick, possibly because of its higher thermal shock resistance. Even such bricks, however, require careful heating at temperatures up to 600°C. if shattering of the brickwork is not to occur due to difference in thermal expansion between the hot and cold parts.

The existence of silicon monoxide, a compound of silicon with only 1 instead of 2 oxygen atoms, is no longer a novelty, but its significance in relation to steelplant operations is still far from clear. In the absence of reducing atmospheres there is little doubt that the silica in silica refractories is present as  $\text{SiO}_2$ , but where conditions are strongly reducing, as they may be for example in, say, an arc furnace, the possibility of silica loss due to the formation of the far more volatile  $\text{SiO}$  cannot be ruled out. Under such conditions certain of the slags commonly assumed as explainable in terms of  $\text{SiO}_2$  type diagrams, may also exist at least in part in a reduced form.

The above summary of the properties of silica is much compressed, and those who wish to go into this rather complex subject in more detail are advised to consult the standard work, *viz.*, Sosman's book on *The Properties of Silica*, together with his 1955 paper to the British Ceramic Society.

#### REACTIONS BETWEEN SILICA AND OTHER OXIDES

The raw materials employed for silica brick manufacture usually show a silica content of between 96 and 98 per cent. Lime is generally added as a bond in an amount varying between 1 and 2  $\frac{1}{2}$  per cent. Smaller amounts of lime tend to give weak bricks both in the dry and fired state, whilst substantially larger amounts adversely affect the properties in service. Fortunately both lime and iron oxide have a relatively small effect on the melting point of silica if present in reasonable proportions. Fig. 36 shows that replacement of as much as 30 per cent. of silica by lime only results in a relatively small drop in melting

point—a phenomenon explained in terms of the immiscibility of the liquids formed by reaction between these materials. The presence of quite small quantities of certain other oxides, notably alumina, destroys this immiscibility, and leads to a steady drop in refractoriness, or an increase in flux content at working temperatures.

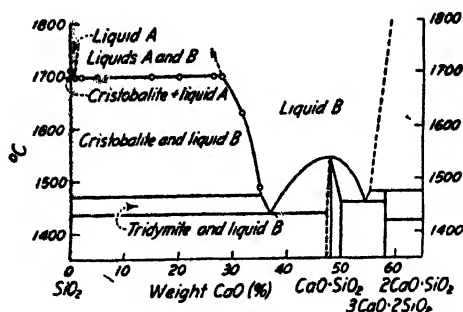


Fig. 36. System  $\text{SiO}_2$ - $\text{CaO}$  (after J. W. Greig).

The reactions between silica and ferrous oxide are shown in the phase diagram, fig. 37. Here again there is immiscibility, and only a small drop in melting point with up to 40 per cent. substitution by ferrous oxide. In actual practice the state of oxidation of the iron is normally higher, but as will be seen later the same tendencies exist. This ability of silica to resist attack by iron oxide is, of course, of paramount importance in the open-hearth furnace, where given adequate temperature control the wear is mainly due to the arrival of iron oxide from the charge and the subsequent dripping away of the reaction product from

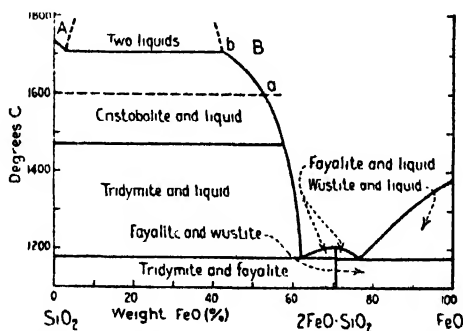


Fig. 37. System  $\text{SiO}_2$ - $\text{FeO}$  (after N. L. Bowen and J. F. Schairer. Revised by R. B. Sosman).

the working face. Sosman suggests that this iron oxide pick-up limits the maximum safe operating temperature to  $1650^{\circ}\text{C}$ . Since pure iron itself melts at  $1535^{\circ}\text{C}$ ., and low carbon heats are frequently tapped at temperatures above  $1600^{\circ}\text{C}$ ., the safety margin would be a small one even if no slag were present. When, however, it is noted that there is often a temperature gradient through the slag of as much as  $10^{\circ}\text{C}$ . per inch, and that as much as 7 in. or 8 in. of slag are often carried, the wonder is that steel can be made at all. The answer lies in design, it being always possible to lower the temperature of the roof with respect to the bath, merely by raising it. In this connection it is particularly interesting to note that the distance between the top of the slag and the highest point of the roof differs relatively little from one furnace to another in spite of great variations in tapping capacity. As regards the actual maximum safe temperature, the most commonly used figure is  $1650^{\circ}\text{C}$ ., though certain plants run at temperatures as high as  $1680^{\circ}\text{C}$ . Recent work on roof temperature distribution shows that to do so certainly calls for both a well designed furnace and a high degree of operating control. Even with the best pyrometers calibration errors of  $10^{\circ}$  to  $20^{\circ}\text{C}$ . are not uncommon, whilst differences between the measuring point and the hottest spot in the furnace roof are often as great as  $30^{\circ}\text{C}$ . Recent research on a hot metal furnace shows that temperatures of over  $1700^{\circ}\text{C}$ . were recorded for short periods on the exit end, even though the central region of the roof was maintained at  $1650^{\circ}\text{C}$ . This marked "out-of-balance" was, however, associated with the evolution of carbon-monoxide from the bath following the hot metal addition.

The reactions of silica with alumina are of the greatest practical importance, since alumina is one of the principal impurities in silica rocks, and is also present in many siliceous cements and patching materials, *e.g.*, those used in facing launders and making acid furnace tapholes. Furthermore most silica cements contain sufficient clay to render them workable with a trowel. Detailed discussion of the equilibrium diagram  $\text{SiO}_2\text{-Al}_2\text{O}_3$  (fig. 117) will be left until Chapter VI, since its greatest importance lies in the light it sheds on a range of alumino-silicate refractories, starting with siliceous firebricks and moving towards pure alumina itself but it should be stressed here that the eutectic (*i.e.*, the lowest melting point in the system) occurs with an alumina content of only 5 per cent. corresponding roughly to 12 per cent. of clay. It will be seen that this composition melts at about  $1545^{\circ}\text{C}$ ., a refractoriness well below that permissible in the operating chamber of a steelmaking furnace. Insufficient appreciation of this simple observation, has led in the past to a great deal of trouble in steelworks, *e.g.*, the use of clay rich silica cements for the setting of

first-grade silica bricks. Such a procedure not only damages the bricks themselves, but may lead to softening and even lubrication of the joints to a point where one course slides readily over the other, and quick deterioration of the structure occurs.

In recent years interest has been focussed on the effect of titania on the melting point of silica, mainly because of its presence in substantial amounts in certain special silica rocks of the chalcedonic type. In this connection the equilibrium system  $\text{SiO}_2\text{-TiO}_2$ , as worked out by Bunting and revised by DeVries, Roy and Osborn, is of interest. It is

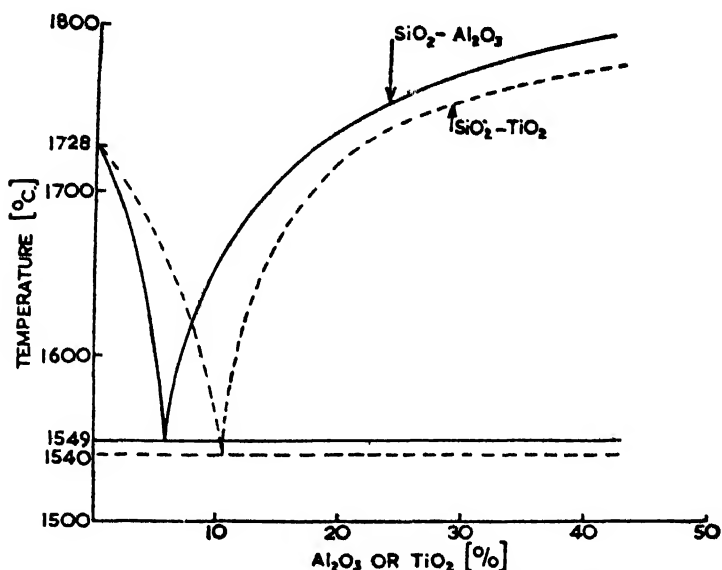


Fig. 38. High Silica end of  $\text{SiO}_2\text{-Al}_2\text{O}_3$  and  $\text{SiO}_2\text{-TiO}_2$  diagrams.

reproduced in fig. 38—superimposed on the alumina-silica diagram. It will be seen from this that whereas the eutectic composition has an even lower melting point with titania than with alumina, the amount of titania needed to achieve it is nearly double the amount required with alumina. It is an extraordinary, and as yet unexplained, phenomenon that low alumina bricks containing natural titania of up to 2 per cent. do not show a low refractoriness either in the laboratory or in the works. Indeed such bricks have given such outstanding performance that attempts have been made—so far without success—to obtain similar properties in other low alumina rocks by adding titania to them.

The binary system  $\text{MnO-SiO}_2$  (fig. 39) is principally of interest to the steelmaker as part of ternary and quaternary systems containing

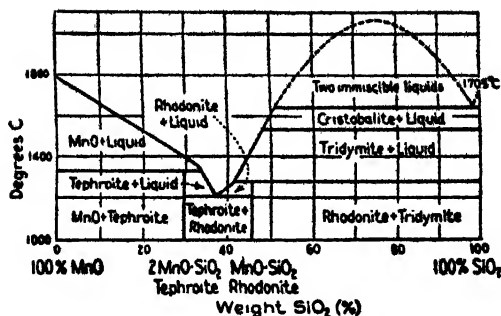


Fig. 39. System  $\text{SiO}_2$ - $\text{MnO}$  (after J. White, D. D. Howat and R. Hay).

these oxides. The ternary systems containing silica that are of most interest to the steelmaker, being:

- $\text{FeO}-\text{Fe}_2\text{O}_3-\text{SiO}_2$  .. fig. 40.
- $\text{CaO}-\text{FeO}-\text{SiO}_2$  .. figs. 41 and 42.
- $\text{FeO}-\text{MgO}-\text{SiO}_2$  .. figs. 43 and 44.
- $\text{FeO}-\text{MnO}-\text{SiO}_2$  .. fig. 45.

The publication by Muan of his work on the system  $\text{FeO}-\text{Fe}_2\text{O}_3-\text{SiO}_2$  marks a major advance in our knowledge of steelplant refractories, since it enables predictions to be made regarding the effect of furnace atmosphere on the resistance of silica bricks to attack by slags high in iron oxide. It confirms earlier opinion that where conditions may be reducing, *e.g.*, in the regions of an open-hearth furnace roof adjacent to the back and front wall, the rate of attack for a given temperature and iron oxide deposition may be appreciably greater than where substantial amounts of free oxygen are present.

Figs. 39-45 are particularly interesting in connection with the formation of acid open-hearth and acid bessemer slags. The  $\text{CaO}-\text{FeO}-\text{SiO}_2$  diagram is valuable, since it enables predictions to be made regarding the attack by iron oxide-lime mixtures on silica under reducing conditions, whilst the  $\text{MgO}-\text{FeO}-\text{SiO}_2$  diagram—apart from its importance in connection with magnesite bricks—illustrates the risk of reaction between such bricks and silica when heated in contact in a furnace whose atmosphere is rich in iron oxide.

Mention should also be made of the studies by Agamawi and White on the system alumina-titania-silica. The authors carried out a preliminary investigation on melting point relations in this system, to determine the cone fusion point of a large number of compositions. The results are summarised in fig. 46, from which will be seen that the lowest melting point found was 1500°C. This work, which was initiated

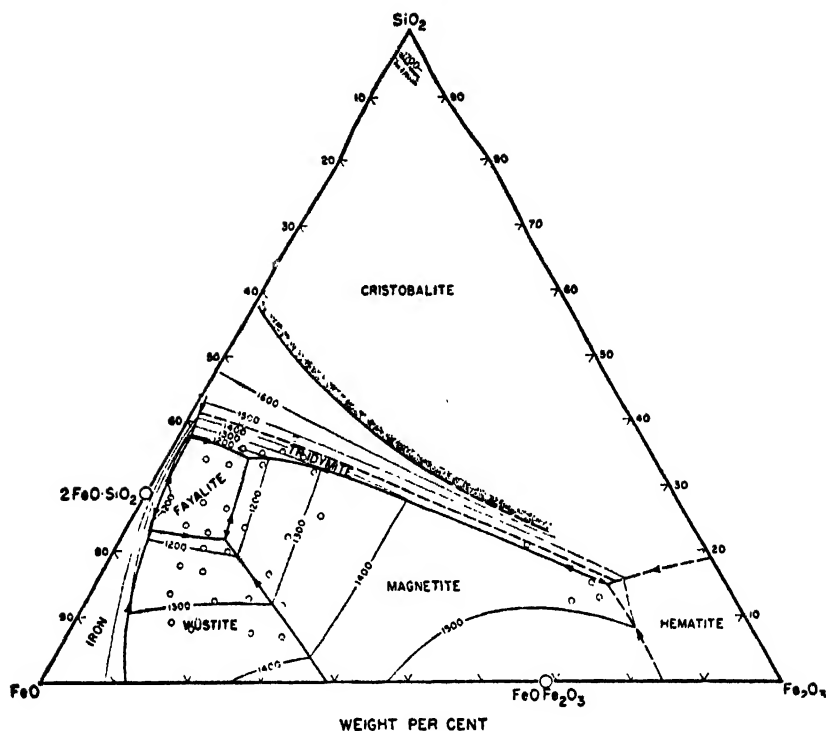


Fig. 40. Phase equilibrium diagram is presented for the system  $\text{FeO}-\text{Fe}_2\text{O}_3-\text{SiO}_2$ . Circles represent compositions, as shown by analysis, of mixtures at liquidus temperatures. Light lines are liquidus isotherms at  $100^\circ\text{C}$ . intervals and heavy lines are boundary curves, with arrows pointing in the directions of falling temperatures. The tridymite-cristobalite boundary curve is dashed. Medium lines with stippling on one side indicate limit of two liquid region (after Muan).

because of the current interest in titania-rich quartzites, was brought to a conclusion by considering four specific compositions:

- (1) 98 per cent.  $\text{SiO}_2$  : 2 per cent.  $\text{Al}_2\text{O}_3$ .
- (2) 98 per cent.  $\text{SiO}_2$  : 2 per cent.  $\text{TiO}_2$ .
- (3) 98 per cent.  $\text{SiO}_2$  : 1.12 per cent.  $\text{Al}_2\text{O}_3$  : 0.88 per cent.  $\text{TiO}_2$ .
- (4) 97.7 per cent.  $\text{SiO}_2$  : 0.30 per cent.  $\text{Al}_2\text{O}_3$  : 2.0 per cent.  $\text{TiO}_2$ .

Composition 3 represents silica containing 2 per cent. of the compound  $\text{TiO}_2-\text{Al}_2\text{O}_3$ , whilst composition 4 can be considered as representing South African silcrete. It will be seen that alumina is roughly twice as effective a flux as titania and that the silcrete composition is only slightly inferior to that containing 2 per cent. titania without alumina.

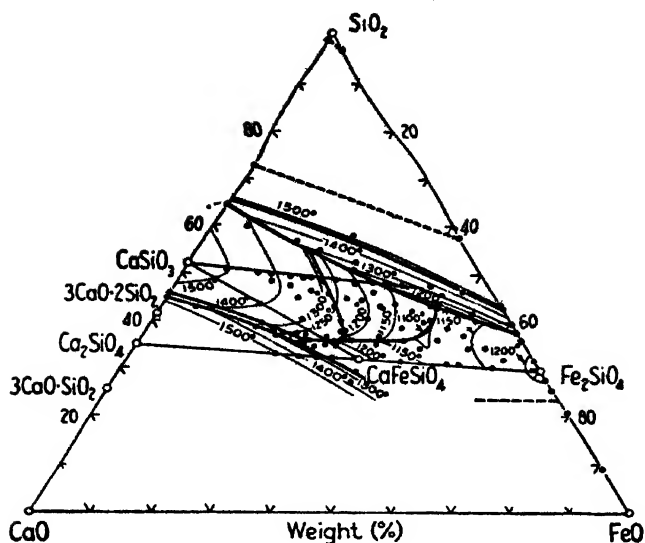


Fig. 41. Isotherms of the system  $\text{CaO}-\text{FeO}-\text{SiO}_2$  (after N. L. Bowen, J. F. Schairer and E. Posnjak).

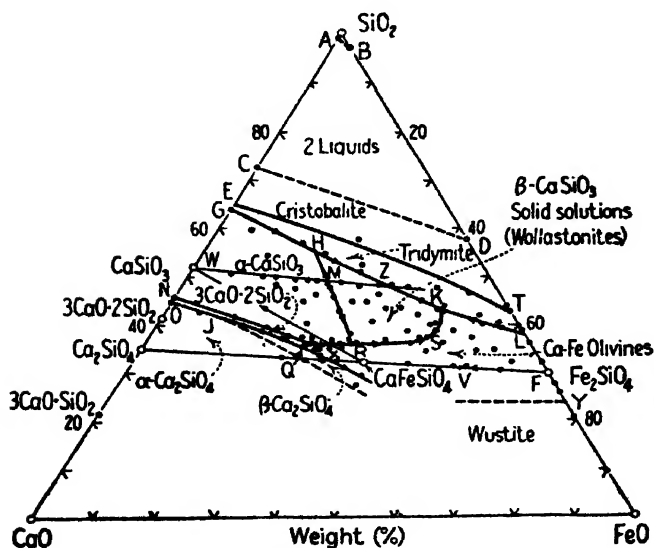


Fig. 42. System  $\text{CaO}-\text{FeO}-\text{SiO}_2$  (after N. L. Bowen, J. F. Schairer and E. Posnjak).



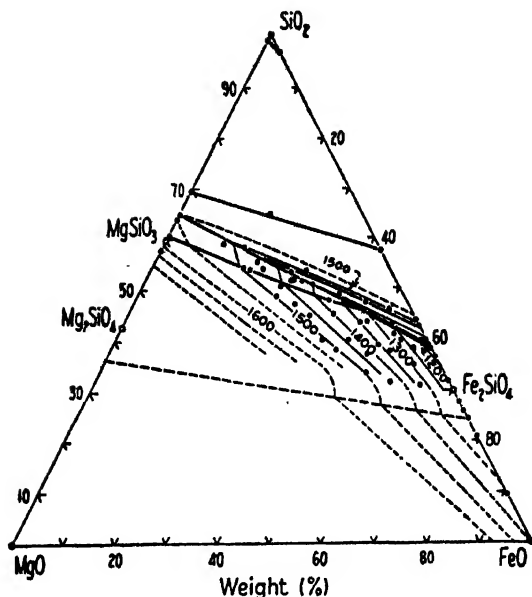


Fig. 43. Isotherms of the system  $\text{MgO-FeO-SiO}_2$  (after N. L. Bowen and J. F. Schairer).

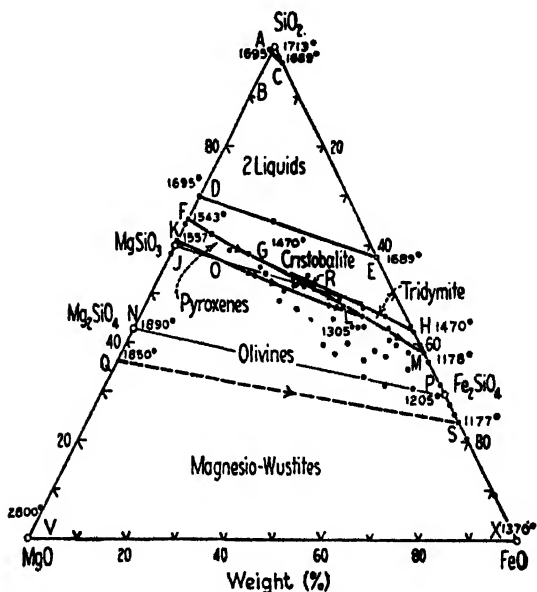


Fig. 44. System  $\text{MgO-FeO-SiO}_2$  (after N. L. Bowen and J. F. Schairer).

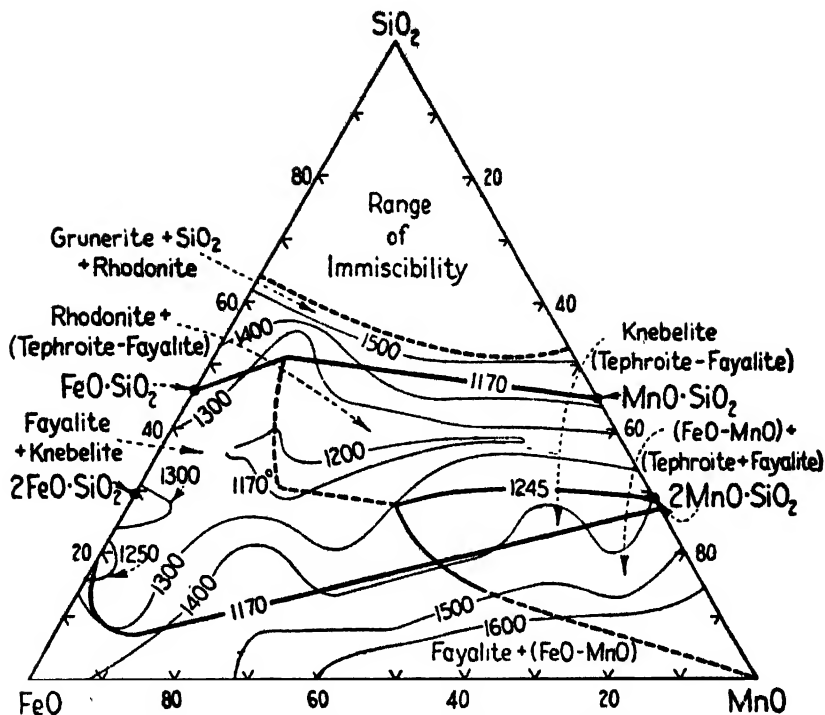


Fig. 45. System  $\text{FeO-MnO-SiO}_2$  (after Maddocks).

The least liquid formation occurred with compositions in which titania and alumina were present in equimolecular amounts.

## RAW MATERIALS

British resources of ganister and silica rock were very fully reviewed by W. Davies in 1948, who assumed that the raw material for the manufacture of high quality silica bricks should contain at least 97 per cent.  $\text{SiO}_2$ , and that of the other constituents  $\text{Al}_2\text{O}_3$  should be less than 1 per cent., and alkalis less than 0.3 per cent. In other words the material should consist essentially of quartz; alkali bearing silicates, such as feldspar, muscovite, and sericite, only being present in small amounts. The distribution of ganister and silica rocks in Great Britain is shown diagrammatically in fig. 47. Davies concluded that the supply of suitable materials in Great Britain was extremely limited, commercial quantities of proved quality quartzite being principally available in certain localities in North and South Wales. Certain

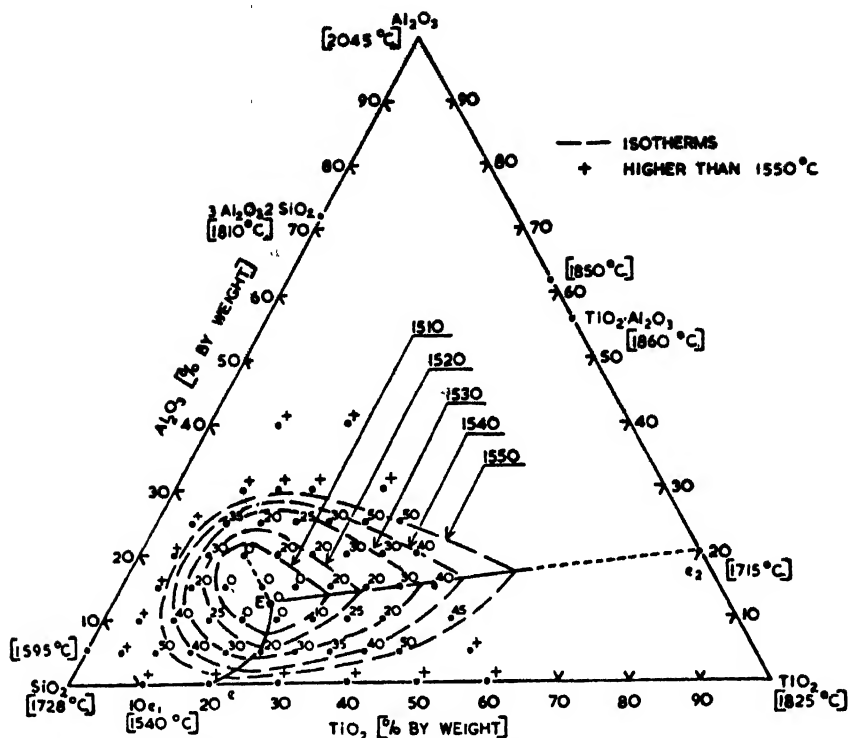


Fig. 46. Cone fusion diagram of system  $\text{Al}_2\text{O}_3\text{-TiO}_2\text{-SiO}_2$ . Figures alongside points show fusion temperatures  $^{\circ}\text{C}$ . in excess of  $1500^{\circ}\text{C}$ . (after Agamawi and White)

Scottish localities were also promising, but had not yet been fully proved. There were still reserves of ganister in the Northern Pennines and in Yorkshire and Derbyshire, but the present costs of mining were excessive. This was also true of the Moor Grit in the North Yorkshire moors. He suggested that the silica brick industry should be based on the following assumptions:

- (a) Progressive exhaustion of the already limited supplies of high quality quartzite. This could be retarded if the highest quality material were reserved for special purposes.
- (b) The increased use of quartzitic sandstones.
- (c) Research developments making possible the use of quartzose sandstone such as those of North Wales and also of high-silica sands.
- (d) The improved mining of ganister, particularly in the Northern Pennines.



Fig. 47. Distribution of ganister and silica rock in Great Britain. Map prepared by Davies on basis of Geological Survey.

Reports of exceptional roof lives obtained in South Africa with bricks made from Mossel Bay silcrete aroused interest (in 1938) in Great Britain, and trials would have been arranged immediately but for the outbreak of war. Following the war and the reported advantages gained from the use of so-called "super-duty" silica bricks in the United States, the whole subject of quartzite and silica brick quality was reopened. Considerable quantities of silcrete material were imported into Great Britain, and as will be seen later, bricks having remarkably good properties were made therefrom. The fact that a marked improvement in performance could be obtained from the material already having a melting point so close to that considered theoretically possible led to a great increase in interest in raw materials, and to the development, particularly in Great Britain, of a considerable number of special silica bricks, possessing not only a low alumina content but also a low porosity.

The significance of these developments can best be understood by comparing the structure, composition and behaviour during firing, of characteristic products. Table IV summarises the observations made on a normal Welsh quartzite, a small sample of Sharon conglomerate from the United States (not necessarily typical of the deposits as a whole), a Findlings quartzite from Germany, and an Opaline silcrete from Albertinia in South Africa, of the general type now employed for silcrete brick production.

It will be seen from the photomicrographs, figs. 48*a*, *b*, *c* and *d*, pages 89 and 90, that the physical structure of these 4 raw materials are all quite different. The magnified sample of the Sharon conglomerate shows only a few crystals, the Welsh quartzite a larger number but still a coarse structure, whilst the Findlings quartzite shows a mixture of coarse and fine crystals, and the Opaline silcrete an almost entirely micro-crystalline condition. The chemical analyses selected are not necessarily typical but the general impression created by them, *viz.*, that of Sharon conglomerate, Findlings quartzite, and Opaline silcrete, are all relatively low in alumina (less than  $\frac{1}{2}$  per cent.), whilst the Welsh material has an alumina content between  $\frac{1}{2}$  and 1 per cent. is probably fair. One intriguing feature of the analysis is that titania, which is negligible in both the Welsh and Sharon material, occurs in quite substantial quantities (between 1 and 2 per cent.) in both Findlings and silcrete quartzites. A further point which is considered by British technologists to be of great importance, is the behaviour of these different materials on firing. The data given, although unfortunately not all obtained under similar conditions, having been done at different times by different operators, does nevertheless show that the increase

TABLE IV  
PROPERTIES OF WELSH, SHARON, FINDLINGS AND SOUTH AFRICAN QUARTZITES

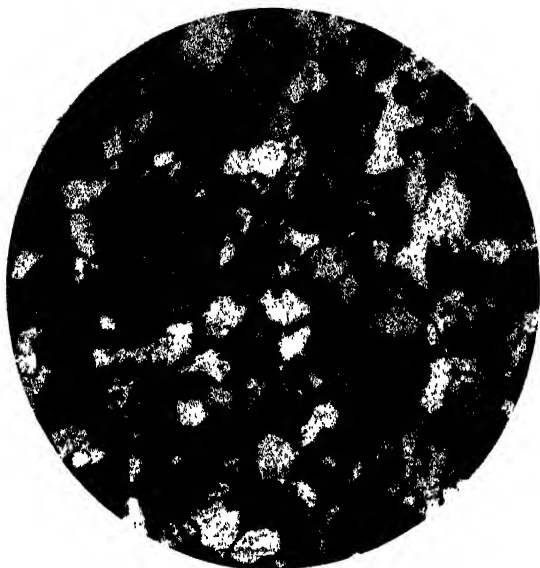
	(1) <i>Coarse-grained quartzite, (washed) Bwlchgwyn, N. Wales</i>	(2) <i>Sharon conglomerate U.S.A.</i>	(3) <i>Chalcedonic silica matrix, high titania content Findlings quartzite Germany</i>	(4) <i>Opaline silcrete Albertinia, S. Africa</i>
Micro examination	Compact tough grey rock composed of interlocked strained quartz grains with some interstitial sericite and kaolinite	Compact translucent pebbles composed of closely sintered quartz grains, often much strained and containing abundant inclusions. Grain size generally 0.1-0.5 mm.	Compact fawn rock breaking with conchoidal fracture. Composed of scattered quartz grains set in an extremely fine-grained chalcedonic matrix	Hard translucent fawn to pale grey rocks breaking with conchoidal fracture: "ring" under hammer and are rather brittle. Composed of a few scattered quartz grains set in a compact matrix of extremely fine-grained transparent chalcedonic silica
Chemical analysis (%) $\text{SiO}_2$ $\text{TiO}_2$ $\text{Al}_2\text{O}_3$ $\text{Fe}_2\text{O}_3$	97.39 0.11 0.73 0.78	98.0 0.1 0.3 0.5 $\text{Al}_2\text{O}_3 + \text{TiO}_2 +$ alkalis 0.5	97.8 — 0.4 0.3	96.1 - 97.8 1.50 - 1.93 0.38 - 0.47 0.16 - 0.56
Physical properties: <i>As received:</i> Specific gravity Porosity (%) <i>After being fired:</i> Specific gravity Porosity (%)	2.66 1.6 2 hour @ 1450°C. 2.37 16.8	2.64 0.6 1 hour @ 1450°C. 2.37 15.8	2.64 0.3 1 hour @ 1450°C. 2.41 3.0	2.62 - 2.67 1.1 - 7.4* 2 hours @ 1500°C. 2.27 - 2.30 3.9 - 9.9*

\* High values due to interstitial cavities and large pores.

in porosity, both with Findlings and silcrete material, is very much less than with Welsh quartzite or Sharon conglomerate. There is no doubt this is a major factor in the outstanding performance of silcrete bricks, and to a less extent of Findlings quartzite, it being possible to produce bricks from these materials having porosities of the order of 16 per cent., a figure extremely difficult to obtain with either Welsh quartzite or American super-duty raw materials.

According to Sosman, silica suitable for brickmaking could probably be found in any county in the United States, though the operation of certain of the materials would clearly be uneconomic. The main American deposits are the Tuscarora formation of Pennsylvania, the Baraboo formation of the Devil's Lake region in Wisconsin, and the Weisner formation of Alabama. The first two mentioned are usually described by the terms Eastern and Western quartzite, it being recognised that bricks made from these materials tend to differ in properties; the Western brick for example having a higher thermal conductivity due to its lower porosity.

X-ray samples taken of different quartzites reveal additional information regarding both grain size and perfection of crystal. The X-ray photographs shown in figs. 49*a*, *b* and *c*, p. 91, indicate that whereas, for example, Sheffield ganister is made up of fairly small uniform crystals, the Findlings quartzite contains a great deal of fine material, which gives rise to the smooth rings on which are super-imposed occasional dots, due to larger grains. With Welsh quartzite the spots are much larger and more irregular. Examination of certain pebbles which had behaved peculiarly when made up in brick form, gave even more surprising results, as is shown by figs. 50*a*, *b* and *c*, p. 92. These particular pictures were taken by a modified Laue method using mixed white and characteristic radiation from a copper or cobalt target. The first photograph (fig. 50*a*) shows what was probably a single crystal that had become fragmented, with the fragments randomly distributed about the mean. In fig. 50*b* the starlike appearance (or "asterism") suggests that there has been a breakdown into fragments whose orientations are more orderly, being related to some specific direction in which the crystal has been distorted. Fig. 50*c* shows a back reflection photograph of the same sample examined by transmission in fig. 50*b*. The precise significance of such photographs is still far from clear, but they at least suffice to show that what may appear under the microscope to be a single grain is either a grain that is highly strained or one that has already broken up into a series of smaller grains having an orientation specifically related to that of the original. These observations are doubtless related to earlier studies made with the microscope, where it



*Fig. 48. (a) Welsh quartzite—Bwlchgwyn.*

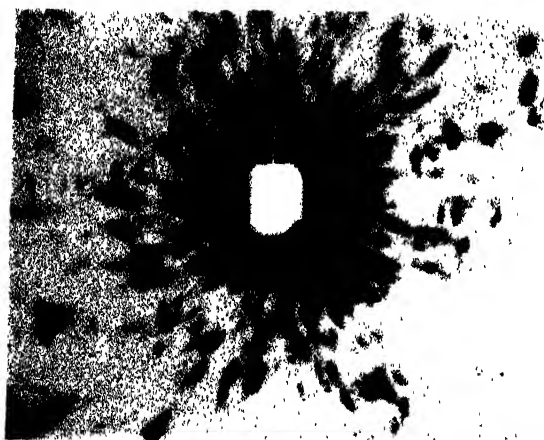


*Fig. 48. (b) Sharon conglomerate—U.S.A.*



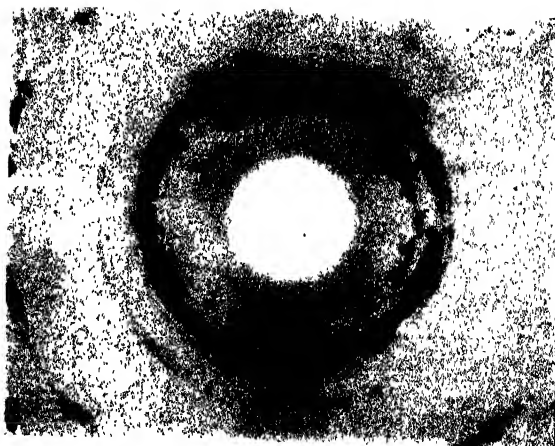
*Fig. 50.*

*(a) X-ray (pinhole transmission) photograph of quartzite pebble indicating fragmented large crystal and random distribution.*



*(b) X-ray (pinhole transmission) photograph as (a) but showing more orderly breakdown of large crystal.*

*(c) As (b) but using back reflection method.*



was found that crystals showing a wavy extinction tended to show a higher conversion rate on firing.

Other materials used in silica brick making are silica sand, flint and crushed "wasters", *i.e.*, damaged bricks. The first is often added to the extent of 20 or 30 per cent. usually as ball-milled fines, whilst the last finds a use for shapes cracked in the kiln and can be of considerable assistance in reducing firing expansion on large blocks.

Long before silica bricks were invented use was made by furnace operators of natural sandstone. Certain specialised types of material, such as the Buckeye quartzite in the United States, continue to be used in the raw state, *e.g.*, for the lining of Bessemer converters, and experiments are being carried out to exploit the use of other natural sandstones of this type. It is too early to state whether they are likely to be in any way competitive with silica, but it has already been demonstrated that in certain positions, *e.g.*, soaking pit walls and acid Bessemer converter linings, they are capable of a performance that makes them competitive with high quality silica or firebrick. The properties of these natural stones vary very considerably from one quarry to another and indeed within a given quarry. A general impression of their properties can be gained from Table V, which gives the results on certain British and American materials. It will be seen that their porosity as mined is lower than that of most silica bricks, and their bulk density considerably higher, due to the fact that they consist essentially of raw quartz. After firing the porosity rises quite appreciably, and the specific gravity indicates conversion to less dense forms. As will be expected from the alumina content, the melting points of such materials are markedly below those of good quality silica bricks, though sometimes higher than those of second quality silica bricks.

## MANUFACTURE OF SILICA BRICKS

Those who wish to make a specialised study of silica brick manufacture, would be well advised to start by reading the contribution by T. R. Lynam in the Ceramics Symposium on which the author has drawn heavily in preparing the present section. According to Lynam, the first silica brick was made by W. W. Young in 1820, who used lime as a bond for silica sand. What is particularly interesting to the steelmaker, is that according to Job Holland, Siemens, in conjunction with Allen, made bricks of the Dinas rock so abundant in the Vale of Neath. Even in these first days of the open-hearth furnace, silica bricks were made by adding a proportion of either clay or lime to the ground rock. This must be one of the first recorded co-operative researches between brickmaker and steelmaker aimed at providing the latter with a brick

TABLE V  
PROPERTIES OF SANDSTONE, FIRESTONE AND SECOND QUALITY SILICA BRICK

	American Firestone	Cumberland Sandstone A	Cumberland Sandstone B	Second quality Silica brick
Chemical analysis (%):				
SiO <sub>2</sub> ..	89.5	90.0	81.5	91.3
Al <sub>2</sub> O <sub>3</sub> ..	3.3	4.1	8.2	3.0
TiO <sub>2</sub> ..	0.2	0.6	0.7	0.1
Fe <sub>2</sub> O <sub>3</sub> ..	2.4	1.5	3.6	1.0
CaO ..	0.4	2.5	2.0	3.0
MgO ..	0.6	1.6	Trace	0.6
Loss on ignition (%) ..	2.0	1.1	3.3	Nil
Apparent porosity (%) ..	20.8	22.4	8.3	24.3
Bulk density, g.p.ml. ..	2.11	2.06	2.40	1.90
Apparent solid density, g.p.ml.	2.66	2.65	2.63	2.51
Refractoriness, °C. ..	1700	1670	1650	1680
Permanent linear change on reheating (2 hours @ 1500°C.) (%) ..	8.9	10.7	—	5.0

that would stand up to the increasingly arduous conditions imposed by the furnace designer and operator. The success of this effort led to similar experiments in the Sheffield district, where a similar rock—ganister—was readily available, and it was Young—a nephew of Allen—who for some years had been grinding ganister at Oughtibridge, who built a small kiln and made the first silica bricks on the site of the present Oughtibridge Company. From then onwards there was a steady development in technique. What perhaps should be stressed was the remarkable insight of Heinrich Koppers, who in 1906—7 years before the publication of Fenner's fundamental work on the inversion of silica—produced a specification which might well be used to advantage even today. It suggested, for example, a maximum lime content of 2 per cent., firing to complete conversion, slow cooling to avoid cracking, freedom from permanent expansion on reheating, and no softening at 3000°F. under a load of 25 lb. per sq. in.

Another major step forward was the demonstration by Hugill and Rees of the marked effect of grading on the porosity of silica bricks.

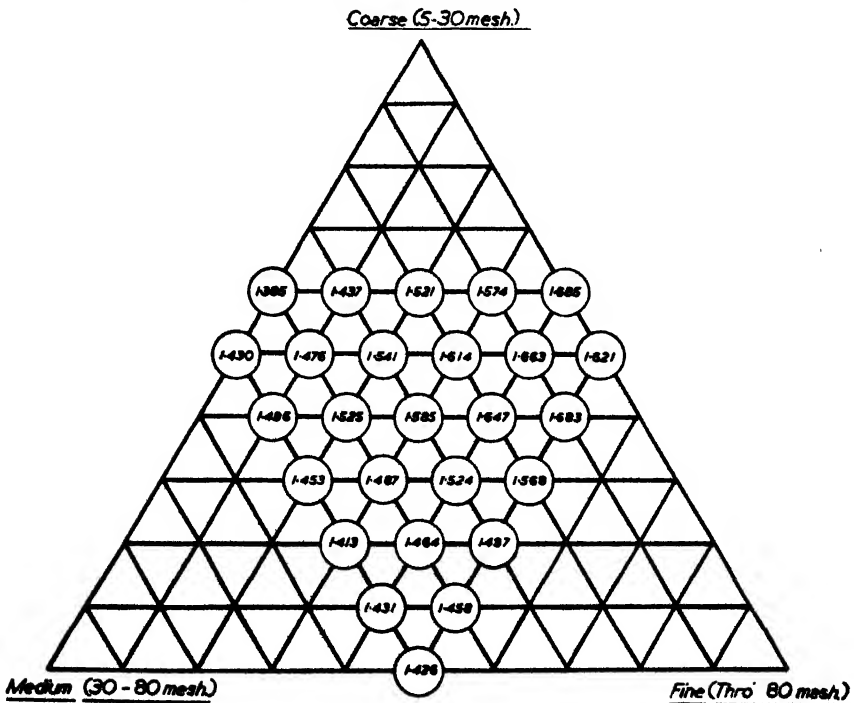


Fig. 51. Influence of grading on the bulk density (g.p.m.l.) of silica bricks (after Hugill and Rees).

Their discovery, illustrated by fig. 51, that a 45 per cent. coarse; 10 per cent. medium; 45 per cent. fine grading gave a high bulk density in the fired product, was the starting point for improvements in many other refractories, *e.g.*, induction furnace linings. These authors also carried out a most useful piece of work on the effect of repeated firing

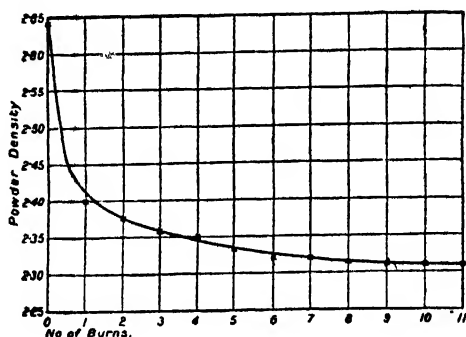


Fig. 52. Influence of repeated firing on the powder density of silica bricks (after Hugill and Rees).

on the specific gravity of silica bricks (fig. 52). Under the conditions employed, the specific gravity fell from 2.65 for the raw material to 2.41 after one firing, 2.38 after 2 firings, and then gradually to a limiting value of about 2.31. They also showed that the porosity

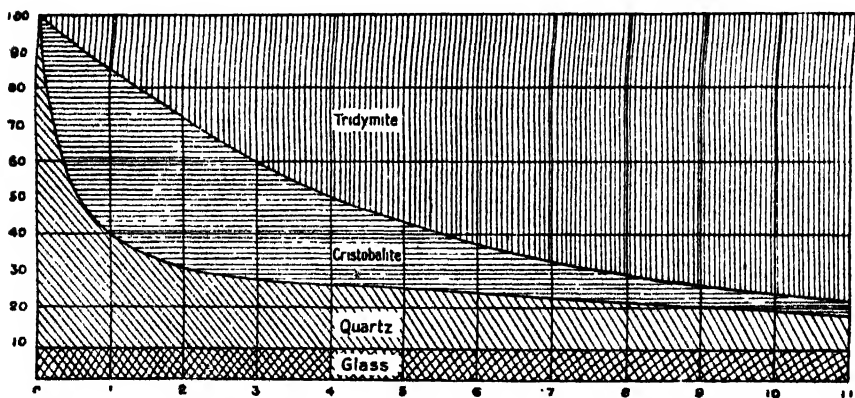


Fig. 53. Influence of repeated firing on the proportions of tridymite, cristobalite and quartz in silica bricks (after Hugill and Rees).

although increasing considerably as a result of the first firing, subsequently tends to decrease. The authors also determined the proportions of tridymite, cristobalite, quartz, and glass (estimated as approximately 10 per cent. throughout), and thus laid the foundation work for the examination of fired silica bricks (*see* fig. 53). The slowness with which the coarser grains are converted is of considerable interest, as is also the gradual replacement of cristobalite by tridymite with the firing temperatures used. As already stated, an enormous amount of work has been done on the effect of different mineralisers on the rate of conversion, and on the percentage of tridymite formed, but strangely enough the present day brick rarely contains any addition other than the originally used lime bond, together sometimes with a small amount of iron oxide. Attempts have been made to produce bricks commercially without added bond, but the results obtained in service did not show sufficient benefit to offset the lower cold crushing strength.

The general procedure at present employed in silica brick manufacture can be summarised briefly as follows:—

#### GRINDING AND GRADING

After washing to remove clay impurities, the stone is passed through a primary jaw crusher, generally yielding a product of about 2 in. size. Samples are often taken at this stage for raw material quality control. The lump material is then recrushed in a disintegrator, cone crusher, or the like, and sometimes elevated to electrically vibrated or mechanically driven screens for separation into grain size groups, any oversize being returned for regrinding.

No matter how good the plant, such dry grinding and screening of silica is liable to create dust hazards, and an alternative method, such as grinding in a perforated ball mill fitted with sieves to give a controlled grading, has much to recommend it. The product of such a mill is usually deficient in fines, but this can be justified by the addition of ball mill ground material so as to get a 45-10-45 type grading. The requisite amount of hydrated lime, mineralisers, and sulphite lye may be mixed with these fines in a revolving-bottom counter-flow type mixer, the rolls of which are raised to prevent further grinding. After a mixing time of, say, 20 minutes, a sample is taken for grading control, determination of free lime content, and moisture.

In earlier days solid bottom pan mills were often used for the batch preparation, and there is no doubt that they are capable of giving an excellently graded batch, and consequently a brick of high bulk density. Their use has, however, been largely abandoned due in part to the difficulties of control when the pan bottom begins to wear.

### MOULDING

The older method of slop moulding has largely gone out, having given way to pressing in hydraulic, toggle type or table type presses, capable of larger production rates and better finish. The fact that the moisture content tends to go down with the making pressure, enables bricks to be moulded that are far less liable to slumping on turning out and, therefore, retain their excellent shape. Special shapes are, however, still made by hand tamping or pneumatic rammers using loose lined moulds.

In order to get good green strength in the brick, sulphite lye, a by-product from the paper industry, is added to the tempering water. This is derived from the non-cellulose portion of the wood by heating with acid calcium bisulphate, the waste liquor being neutralised by lime or soda. A remarkable increase in dry strength can be obtained even with  $\frac{1}{2}$  per cent. of this bond, partly because it tends to travel to the exposed faces on drying, giving a case-hardening action. With large shapes, however, this migration can be an embarrassment, as it may lead to localised cracking if the skin forms before all the moisture has been removed from the centre of the brick.

### DRYING

Some shapes are still dried on hot floors but for the most part this procedure, which often leads to cracking—not revealed until the bricks have been fired—has been replaced by tunnel drying, using steam or waste heat from the kilns. In general the moisture content is reduced to 0.2 per cent. or less before stacking in the kilns.

### FIRING

Dale illustrated his ideas on the changes during the firing of silica by the rather ingenious diagram reproduced here as fig. 54. As Lynam in the Jubilee Symposium on Ceramics states, Dale's work has not received the attention it merits, possibly because it was published under a rather specialised title. Briefly what Dale stated was that: "At approximately 750°C., the unfired brick consists of granules of quartz, separated by a groundmass or matrix containing essentially lime, quartz flour, and the smaller grades of quartz particles (*see fig. 54A*).

Above 800°C., the interaction between the lime and the free quartz surfaces commences. For simplicity, this reaction was regarded as one of solution, resulting in the formation of a lime-silica glass, the silica content of which is continually increasing as time proceeds and temperature rises. This glass can, however, devitrify and precipitate

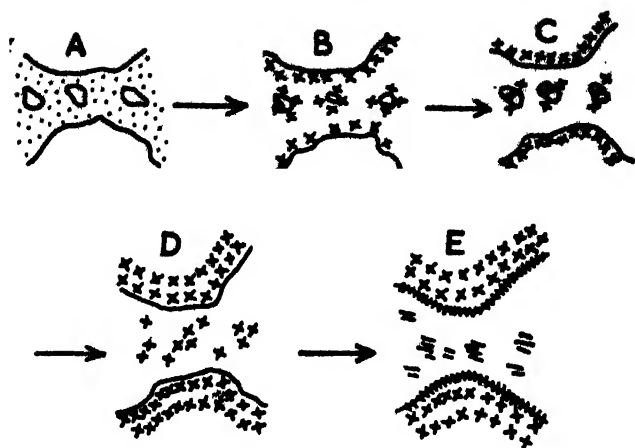


Fig. 54. Changes in silica brick during firing (after Dale).

cristobalite below  $1250^{\circ}$  to  $1300^{\circ}\text{C}$ . in local regions of high silica content (see fig. 54B).

At a temperature above  $1200^{\circ}$  to  $1300^{\circ}\text{C}$ ., the surface of the quartz particles commences to convert directly to cristobalite, and the velocity at which this conversion proceeds inwards increases with further rise of temperature (see fig. 54C). Thenceforward, with rise of temperature, cristobalite dissolves in the lime-silica glass of the groundmass.

As the temperature rises towards the soaking temperature, the conversion of the larger quartz granules is proceeding towards the cores. Small particles of quartz in the groundmass are completely converted, and the silica content of the lime-silica glass is still increasing rapidly by solution of minute cristobalite granules and the neighbouring surfaces of the larger grain (see fig. 54D).

During the soaking period, the lime-silica glass of the matrix becomes saturated at a rate dependent, amongst other factors, on the actual temperature, which is critical. At some degree of saturation, the glass commences to be devitrified, and precipitates silica in that crystalline form called "tridymite". This phenomenon of devitrification naturally lowers the degree of saturation of the glass; more cristobalite dissolves and the reactions cristobalite  $\rightarrow$  saturated glass  $\rightarrow$  tridymite, proceed continuously (see fig. 54E)".

Tests made by Lynam in 1936, in which samples were drawn from a kiln at temperatures of  $1200^{\circ}\text{C}$ . and upwards, gave results that were in good general agreement with the above theory.



The later work described above by Hugill and Rees on repeated firing related to one particular raw material. In practice the manufacturer has often to consider several raw materials and the relative rate at which they invert becomes of extreme importance. When new materials are to be used control trials are normally carried out, the specific gravity at various stages during the firing being determined in

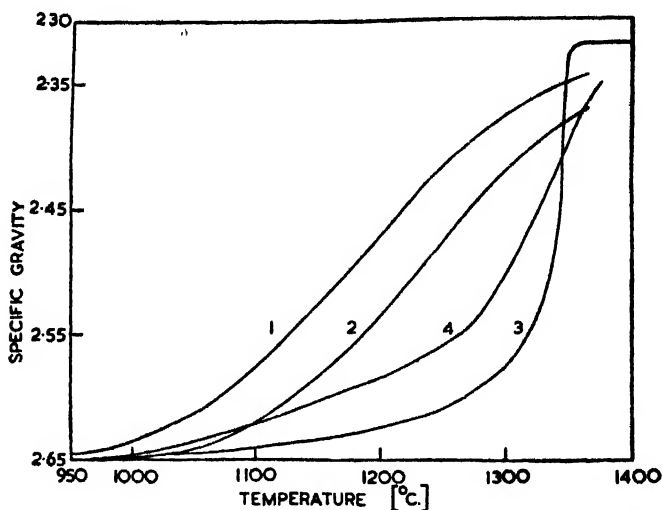


Fig. 55. Inversion-temperature relations for various silica rocks:

- (1) Silcrete.
- (2) Welsh quartzite.
- (3) Chalcedonic type.
- (4) Coarse-grain quartzite of high purity.  
(after Lynam).

the manner shown in fig. 55. This illustrates why brick manufacturers are concerned with other factors than chemical analysis. Sample 1, which is a finely grained silcrete, comes close to being ideal, whilst sample 2 is a coarse grained quartzite from North Wales. Sample 3 shows the almost explosive violence with which a chalcedonic type rock can invert, whilst No. 4 shows the behaviour of a coarsely grained and very pure quartzite.

One of the great difficulties in firing silica bricks is the maintenance of even temperature distribution, and from this standpoint round-down-draught kilns are normally preferred to the rectangular type. Mendheim kilns have also given satisfactory service, whilst at many of

the newly built plants tunnel kilns are employed. As with every other application, this type of kiln tends to suffer from lack of elasticity, and must be of a length to give the necessary heat treatment. Such kilns have, however, been in use for sometime, *e.g.*, at Koppers in Germany, and Harbison-Walker in the United States, and more recently were installed in a new British works at Landore (*see* paper by D. Jones).

Periodically some steel firm becomes greatly excited about the peculiar mottling sometimes observed with silica bricks. This phenomenon which may be due to the presence in the kiln of sulphur-rich moisture from a wet fuel, or the setting of insufficiently dried bricks, does not appear to be of more than academic interest, no evidence having been produced to show that it has any effect on durability in service.

#### QUALITY CONTROL

Periodic gradings are carried out on the batch from the mixer, *e.g.*, on + 30, - 30, + 60 and - 60 B.S.I. mesh basis. Moisture content may be determined by the rapid calcium carbide method in which a weighed sample of batch is placed in a previously calibrated vessel containing calcium carbide. On shaking up, acetylene is liberated, creating an indicated pressure on a dial. The hydrated lime addition is checked by titration. With special bricks, *e.g.*, for open-hearth furnace roofs, bulk density is so important that routine weighings of representative samples taken after moulding or drying are carried out. Firing treatment is generally checked by specific gravity determinations, the procedure to be adopted having been the subject of considerable study, *e.g.*, by Hale, who evolved a sampling plan for use in a particular works. In the recommended procedure, 4 bricks are taken from each consignment, the specific gravity of each being measured, and the average of the 4 determined. If less than 2.365 the load is accepted, if 2.365 or more the load is rejected. It is stated that this plan assures these particular makers that loads with 1 per cent. of bricks having a specific gravity of over 2.40 will be accepted, with a 5 per cent. (1 in 20) risk of wrongful rejection, whilst it assures the user that loads with 10 per cent bricks of specific gravity greater than 2.40 will be rejected with a 5 per cent. (1 in 20) risk of wrongful acceptance. This plan, which marks a big advance over the usual spot samples, has been fairly widely adopted in the British steel industry.

#### PROPERTIES OF SILICA BRICKS

Some silica bricks have always been better than others, and indeed classification into first and second grades was not uncommon. The

position has now been improved, but complicated by the development of the so-called "super-duty" silica brick. According to the patent by Harvey and Birch (1944) the sum of the percentages of alumina, titania and alkalis in such a brick should not exceed 0.5 per cent. The need for such a low flux contents brick was clearly demonstrated by Kraner, using a split roof technique. The observation, that even a drop of, say, 0.3 per cent. in the alumina content of a brick, was well worth striving for has had a very remarkable effect on silica brick production in Great Britain, leading for example to the development of at least half-a-dozen low alumina type bricks. The principal difference between the British or South African bricks, and those produced in America, lies in the porosity, which in turn is a function of the raw material employed. Incidentally the titania content of the silcrete bricks is quite substantial—approaching 2 per cent., and it would therefore appear extremely doubtful whether the titania content should be included in the total of deleterious fluxes.

There has been a natural tendency among silica technologists to think in terms of the most difficult and critical positions, *e.g.*, the roofs of open-hearth furnaces. One consequence of this is that whereas quality in these positions may be assured, it may be relaxed on normal squares, and as a result difficulties experienced in other parts of the furnace, *e.g.*, slag pockets, due to the use of bricks of unusually high porosity and flux content. Actually improved bricks will also be required for these positions since faster driving generally means higher waste gas temperatures.

#### POROSITY

The production of dense silica bricks is by no means easy, mainly because the conversion of the raw material to a less dense form during firing upsets the original arrangement of the grains. It will be seen for example from Table VI, which lists the properties of selected open-hearth roof bricks of normal quality, that figures of 22 to 26 per cent. are typical. Certain types of super-duty bricks (*see* Table VII) show even higher porosities, presumably due to the low flux content and consequent absence of compensating shrinkage. Others, notably bricks made from South African silcrete, show porosities well below the 20 per cent. level.

#### BULK DENSITY AND TRUE SPECIFIC GRAVITY

If bricks are hard-fired, the bulk density is directly related to the apparent porosity. If on the other hand the bricks are soft-fired, then relatively high bulk densities will be obtained. With silcrete type

TABLE VI  
PROPERTIES OF OPEN-HEARTH FURNACE ROOF BRICKS—NORMAL QUALITY

Code No.	British				American		German	
	S.8	S.6	S.5	S.7	S.9	S.2	S.3	S.4
Apparent porosity (%) ..	25.4	22.7	22.8	23.6	26.5	25.8	23.5	20.7
Bulk density—g.p.m.l. ..	1.73	1.79	1.83	1.76	1.75	1.72	1.78	1.85
lb. per cu. ft.	108	112	114	110	109	107	111	116
True specific gravity	2.30 <sub>s</sub>	2.33	2.36 <sub>s</sub>	2.32	2.39	2.30	2.32	2.325
(Rees-Hugill flask)								
Permeability to air (perp. to working face and through one skin), c.g.s. units ..	0.079	0.049	0.041	0.083	0.105	0.189	0.138	0.017
Permanent linear change on reheating (2 hours @ 1500°C.) ..	0.0%	1.1%	0.5%	0.5%	1.7%	0.2%	0.4%	0.4%
Refractoriness ..	1710°C.	1710°C.	1710°C.	1730°C.	1690°C.	1710°C.	1710°C.	1730°C.
Refractoriness-under-load maintained at 1600°C. for 1 hour, 25 lb. per sq. in. Resistance to thermal shock (450° test) ..	0.6% deformation	0.7% deformation	Sheared in 24 minutes	No deformation	Sheared after 7 minutes	Sheared after 30 minutes	1.4% deformation	0.4% deformation
Microscopical examination:	2	2	18	2	3	3	1	8
Approx. % quartz ..	0	30	30	20	30	0	5	20
" " tridymite ..	50	20	35	40	10	50	20	30
" " cristobalite ..	50	50	35	40	60	50	75	50

TABLE VII  
PROPERTIES OF OPEN-HEARTH FURNACE ROOF BRICKS—"SUPER DUTY" QUALITY

Code	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Apparent porosity (%)	20.5	20.7	17.2	29.5	23.7	24.4	25.4
Bulk density (g.p.m.l.)	1.87	1.83	1.93	1.68	1.77	1.75	1.74
(lb. per cu. ft.)	117	114	121	105	110	109	109
Apparent solid density (g.p.m.l.)	2.32	2.31	2.33	2.37	2.32	2.32	2.33
Permeability to air, perp. to working face, 1 skin (c.g.s. units)	0.06	0.05	0.03	0.11	0.21	0.71	0.04
Permanent linear change on reheating (2 hours @ 1500°C.)	0.4	0.4	0.2	3.0	0.4	No change	0.3
Refractoriness-under-load, 50 lb. per sq. in., maintained at 1600°C. for 1 hour	No deformation	No deformation	No deformation	2.0% exp.	—	0.4% def.	0.4% def.
Thermal shock resistance—max. safe heating rate °C./min.	4/5	4/5	3/4	—	—	—	—
Refractoriness, °C.	1710	1710	1710	1710	1730	1710	1710
Cone:	>32	>32	>32	>32	33	>32	>32
Chemical analysis (%):							
SiO <sub>2</sub>	96.6	96.8	94.8	96.0	96.55	94.55	94.50
Al <sub>2</sub> O <sub>3</sub>	0.19	0.49	0.37	0.33	0.39	0.15	0.59
Fe <sub>2</sub> O <sub>3</sub>	0.72	0.70	0.65	0.64	0.88	0.56	0.80
CaO	1.60	1.59	2.07	2.31	2.05	3.16	2.77
MgO	0.07	0.11	trace	0.13	0.03	0.72	0.25
TiO <sub>2</sub>	0.01	0.02	1.70	0.11	0.07	nil	0.51
Na <sub>2</sub> O	0.12	Not determined	0.10	Nil	Not determined	Not determined	Not determined
K <sub>2</sub> O	0.05	0.16	0.16	Nil	—	—	—
Loss on ignition	0.22	—	0.19	—	—	—	—

product, bulk densities as high as 1.95 g.p.ml. are obtained even with a fully converted product.

The true specific gravity, as determined by a pycnometer, differs only slightly from the apparent solid density as determined from the porosity test, and is generally of the order of 2.31 to 2.33 for a fully converted product, ranging up to 2.65 for the completely raw material.

#### PERMEABILITY

Permeability tests are still carried out on silica bricks for use in steelplants, though they do not at the moment appear to add appreciably to the quality assessment. Their inclusion is mainly justified on the grounds that it may at some future date be possible to assess the relation between this property and performance in service.

#### PERMANENT LINEAR CHANGE ON REHEATING

Really soft-fired silica bricks can show an after expansion of several per cent. if refired in the standard manner. If such an expansion occurs in a furnace structure it may well lead to serious distortion apart from the risk of spalling due to nipping. Hard-fired products on the other hand show very little expansion, and the rise say in an arch can, therefore, readily be predicted.

#### REFRACTORINESS

Pure silica is stated to melt at 1723°C., but cone refractoriness tests frequently give fail points as high as 1730°C. The anomaly is explainable partly by different definitions of melting, and partly by the fact that silica skeletons are frequently sufficiently rigid to maintain their shape for some time even though saturated with fluxes. That refractoriness is a serious limitation with silica bricks is shown by the substantial improvement in roof life obtained in plants where roof pyrometry has been well developed and strictly used. The chances of local overheating are thus greatly reduced though by no means eliminated, since imperfect temperature distribution due either to poor design or to flame deflection by scrap may easily result in a particular area being 30° or 40°C. hotter than the control point. Incidentally it should be noted from Tables VI and VII that there is very little to choose between the cone refractoriness of super-duty and ordinary silica bricks.

#### REFRACTORINESS-UNDER-LOAD

Kraner and others have demonstrated that the ability of a silica brick to carry a load at a given temperature is very much dependent on the

liquid content as calculated from equilibrium considerations. Both ordinary and super-duty silica bricks are, however, very good in maintaining a typical arch load of, say, 25 lb. per sq. in. at 1600°C., no deformation being observed with many of the super-duty type bricks and very little with ordinary first-quality material. If, however, the content of alumina or alkalis is allowed to rise to, say, 1½ or 2 per cent., then a very serious deterioration in refractoriness-under-load occurs, failure occurring below 1600°C. even in the rising temperature test. The use of such bricks in roofs might well lead to trouble, and has even been associated with premature failure in slag pocket arches, where the temperature will normally be 100°C. lower than in the main roof.

#### THERMAL SHOCK RESISTANCE

If the small prism test, as developed for other refractories, is applied to silica bricks, even the first reversal generally causes failure. If on the other hand the test temperature is lowered to 450°C. certain bricks (see Table VI) may last as long as 18 reversals. The lack of any close correlation between results of this test and service in the open-hearth furnace, led Howie to develop a new type of test, in which a 3 in. × 3 in. × 4½ in. testpiece is heated on one 3 in. × 3 in. face by means of an electric hotplate. The cobblestone type spalling which occurs is very like the effect observed in an actual furnace when the rate of heating used is excessive. Far more differentiation is obtained using this method than when the testpiece is heated from all sides. Howie found that under these conditions a heating rate of more than 5° to 6°C. per minute resulted in cracking. He also confirmed that soft-fired bricks had a much higher resistance to thermal shock than hard-fired ones. The value of soaking in creosote or tar to reduce spalling in hot repairs was shown to be due to its influence on the rate of temperature rise, the heat needed to evaporate the creosote reducing the rate of temperature increase in the region of the critical cristobalite change.

Later, and as yet unpublished work, shows that if full bricks are used on a hot plate instead of testpieces, the differentiation previously obtained, *e.g.*, the superiority of the Findlings type product, is less marked, the only serious difference between first-quality bricks being that the maximum safe rate of heating is greater with soft-fired than with the hard-fired material.

#### MICROSCOPIC AND X-RAY EXAMINATION

The use of thin sections or of crushed samples of products enables a rapid estimate to be made of the residual quartz content. Differentiation

between cristobalite and tridymite is much more difficult and is best achieved by X-ray powder methods. A rough idea may be gained of the probable permanent linear change in service from the raw quartz content, but the refiring test itself still provides a more reliable answer due to the effect on this property of the type of raw material used in brick production.

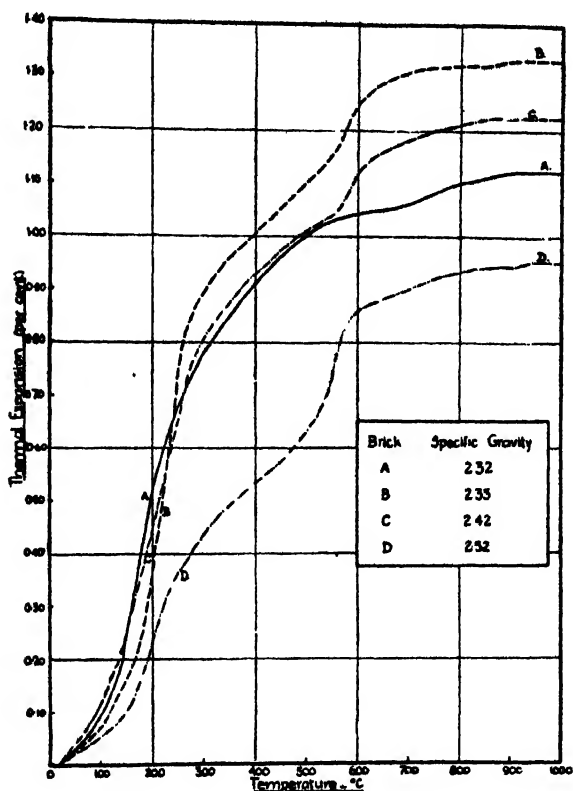


Fig. 56. Thermal expansion curves for silica bricks of varying degrees of conversion (cf. specific gravities).

#### REVERSIBLE THERMAL EXPANSION

The curve shown in fig. 56, may still be considered characteristic of silica bricks of varying specific gravity, and largely explainable in terms of the original work of H. Le Chatelier (see fig. 57). The relatively linear expansion of the soft-fired brick "D" agrees well with the observed higher thermal shock resistance, whilst it will be seen that with certain of the harder fired bricks the expansion between 200° and



300°C. is of the order of  $\frac{1}{2}$  per cent. All the samples except brick "A" show the same inversion at 575°C., corresponding to the alpha-beta quartz change. Incidentally the precise temperature range over which the alpha-beta cristobalite change takes place varies considerably with the raw material used. For this reason certain manufacturers prefer to use a blend of silica rocks.

#### THERMAL CONDUCTIVITY

A typical figure for silica bricks at a mean temperature of 1000°C. is 10 B.Th.U., as compared with, say, 6 to 8 for fireclay bricks, and 12 or over for chrome-magnesite or magnesite bricks. Expressed in terms of service conditions this figure suggests that a 12-in. thick roof with an inside temperature of 1650°C. would have an outside temperature of the order of 250°C.—a result approximately in agreement with practice.

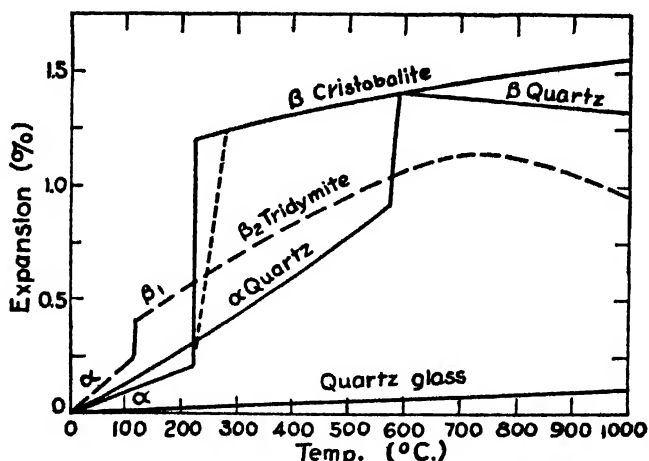


Fig. 57. Thermal expansion of silica minerals (after Le Chatelier).

#### SLAG RESISTANCE

The unusual ability of silica to resist attack by iron oxide and lime has already been mentioned. As these are the main steelplant fluxes, its durability even at temperatures close to its melting point is at least partially explained. Where conditions are strongly reducing, *i.e.*, the flux is FeO rather than  $\text{Fe}_3\text{O}_4$ , attack may proceed more rapidly, whilst relatively small amounts of other corrodents may have a proportionately greater effect. Thus, in the tops of checker chambers,

silica is gradually being replaced by high quality fireclay bricks, or even the 50 per cent. alumina type, due in part to its inability to stand up to the various fluxes which frequently include such things as zinc oxide. A good idea of the probable resistance of silica to different fluxes can be obtained by a study of the equilibrium diagrams reproduced herewith, though it should be remembered that these only refer to *equilibrium*, a condition which rarely obtains.

One recently observed cause of deterioration with silica has been traced to the use of tar impregnation to reduce spalling during hot patching. It has been fairly definitely shown that under certain conditions, presumably those in which the carbon is not rapidly burnt out, a frothing of the brick surface can occur, possibly due to the formation and volatilisation of silicon monoxide within the plastic mass.

#### ACTION OF WATER VAPOUR

A series of experiments by Dodd has shown that certain silica bricks when heated in water vapour at only 1600°C. show marked crumbling and even fusion. The cause of this breakdown is not clear, but the work at least supports the generally held opinion that strongly reducing conditions such as may result from the use of fuel rich in hydrogen (*e.g.*, coke oven gas) may lead to premature failure of open-hearth furnace roofs. The position is however still uncertain, more recent work by Harvey not appearing to check with that reported by Dodd. In this later work bars cut from silica bricks showed no bending or softening after 5 hours in water vapour at 2900°F. (1593°C.).

#### APPLICATIONS

The main usage of silica bricks in steel plants is still in the basic and acid open-hearth furnace (*see* Chapters VIII, IX, and X), though considerable quantities are also used in the manufacture of acid bessemer and electric steel (*see* Chapters XI and XII). Although coke ovens and gas works do not come within the scope of the present text, their usage of silica brick is also substantial and does influence the steel plant, in that similar raw materials are mined for both purposes. Since the life of a coke oven wall does not appear to be critically related to the alumina content of the brick there would appear to be a strong case for reserving low alumina materials for steelplant use.

#### SEMI-SILICA

Considerable use is made in British steelplants of a semi-silica brick containing approximately 88 to 93 per cent. of silica, and made either

from a natural sand-clay mixture or artificially prepared by mixing suitable constituents. The properties of such so-called semi-silica bricks are set out in Chapter IX, where their use in open-hearth furnace checker settings is discussed. It will be seen that their properties are intermediate between those of silica and fireclay. Apart from their relative cheapness, they possess marked advantage over fireclay bricks of freedom from after-shrinkage and over silica bricks of relatively high thermal shock resistance. Perhaps their most useful property is, however, a tendency to glaze readily when used for example in coal-fired reheating furnaces. A peculiarity in their behaviour is the extraordinary way in which slag attack is limited to the surface, thus cross-sections through the semi-silica and silica checker bricks, given in figs. 185 and 186, p. 424, show with silica complete penetration to the centre, whilst with the semi-silica fracture shows a completely white and unaltered core.

This peculiarity of semi-silica bricks has been discussed at some length in a recent paper by Remmey, who deals with the American (New Jersey) type semi-silica, whose silica content is considerably lower, *viz.*, approximately 76 to 78 per cent. He suggests that the proportions of alumina to silica are sufficiently close to those of the eutectic that even a small pick-up of fluxes results in the surface being converted to a glass, whose viscosity is extremely high, due to its high silica content, and so is incapable of being readily absorbed by the fine pores of the unaltered brick. Remmey goes further and suggests that subsequent attack by more fluid slags, high, say, in iron oxide, results in a surface layer of relatively fluid material, which is consistently separated from the body of the brick by the extremely viscous original product.

## SILICOSIS

According to Lynam, discussion with various persons actively engaged in the silica industry in its earlier days, suggests that the main difference between manufacture then and now, lies in the cleanliness of the plant. In the old days it was not at all uncommon for shed floors to be 2 in. to 3 in. deep in silica dust, whilst this same material was used to coat moulds in order to prevent the wet batch sticking to them. The position changed rapidly after the Workmen's Compensation Act in 1919, which led to a far higher standard of cleanliness, to the installation of dust removal and dust laying equipment, and to the use of sawdust or washed natural sand for the dusting of moulds. For some time it was thought that the cause of silicosis was purely physical, the lungs of the victim being lacerated by the sharp particles of silica. More recent theories suggest a chemical mechanism, but the precautions to be

taken remain the same, it being proved beyond doubt that frequent exposure to dust high in silica, whether produced by grinding bricks or crushing quartzite, is highly dangerous. Considerable protection can be afforded by the use of respirators, but wherever possible the nuisance should be stopped at its source, *e.g.*, by changing the methods used in grinding and mixing. It has long been realised that even a small amount of moisture greatly reduces dust formation, and where this can be employed without serious interference with the process it should certainly be adopted. Thus, with acid induction furnace linings, a small amount (1 per cent.) of moisture has no influence on coil insulation but quite effectively lays the dust.

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### *Chapter III*

## MAGNESITE

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CHESNY'S SUGGESTION IN 1936, that refractory magnesia could be produced from sea-water at a price competitive with the mined product, seemed unduly optimistic even to those who knew that he proposed to double the yield by using calcined dolomite as the precipitating agent. Yet by 1953 more than 250,000 tons per annum of sea-water magnesia were being produced, as compared with approximately 380,000 tons per annum of dead-burned Austrian magnesite. Virtually the whole of the magnesite used for refractory purposes in Great Britain now comes from the North Sea at Hartlepool and the adjacent dolomite quarries at Coxhoe.

Even if the hydroxide could be produced at an economic figure it was still by no means certain that it could be made into good bricks. The technologists concerned were, for example, aware of the inability of the silica brickmaker to produce a sound product from 100 per cent. of clean sand, even though the chemical analysis was considered satisfactory. The first comparison of the properties of magnesite bricks (or rather briquettes since they were only 1 in. high) made from sea-water and Austrian magnesite, was carried out at the Central Research Department of The United Steel Companies Limited at Stocksbridge in 1936. The results were so encouraging that those concerned decided forthwith to investigate the possibilities of full-scale production. Since that time natural magnesite has been replaced in one product after another by sea-water magnesia, and with highly satisfactory results, one of the latest developments being a record roof life of over 2000 heats on an all-basic furnace built of chrome-magnesite bricks, the magnesia for which came from the sea.

It would, however, be unfair to give the impression that the change-over was easy. Many difficulties were experienced, both by the producers, *e.g.*, that of getting a sufficiently low lime content, and by the users, *e.g.*, in induction furnaces, where numerous breakouts occurred before a satisfactory lining for a 2-ton furnace based on sea-water magnesia was developed.

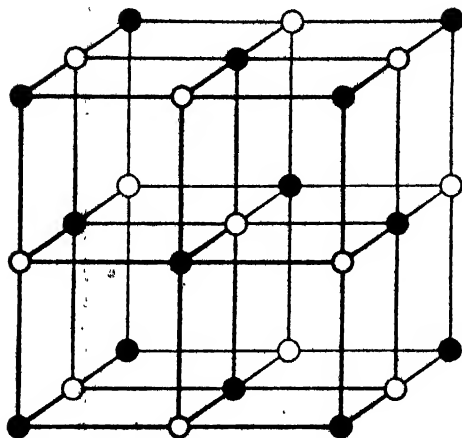
The possibility of extracting magnesia both from dolomite and from brines has interested chemists ever since Davey discovered magnesium metal in 1808. Much attention has been given to the decalcification of dolomite; Pattinson being granted a patent in 1841 for the carbon dioxide process, that still bears his name. By the end of the century many other processes for the extraction of magnesia from dolomite and brines had been proposed and patented, whilst magnesia had been recovered on a small scale from sea-water using lime as a precipitant at Aigues Mortes on the Mediterranean coast of France—see literature survey by G. E. Seil.

The first large scale production of synthetic magnesia was by the California Chemical Company in 1931, using sea-water bitterns, *i.e.*, the liquor remaining after the production of salt by solar evaporation. For lime use was made of oyster shells. At roughly the same time the Marine Chemical Company made use of synthetic magnesia for pharmaceutical purposes. The first commercial plant for refractory magnesia was, however, built by the Steetley Company in 1938 in association with H. H. Chesny. The emphasis given above to this new development should not be taken to mean that it eclipses the production of refractory magnesite from the other raw materials mentioned later. These are still the most important source and possess properties which make them peculiarly suited for certain purposes. Even so the production of sea-water magnesia is one of the outstanding developments in refractories in the first half of the 20th century.

## MAGNESIA—THE COMPOUND $MgO$

### ATOMIC STRUCTURE

Although complex in its behaviour as a refractory, magnesia is fundamentally the simplest of all the materials used in furnace construction. The photograph reproduced as fig. 58, p. 121, shows a magnesia crystal over 1 in. in length and perfectly transparent that was produced accidentally by the Norton Company as a result of prolonged heating in an electric furnace. It is so perfect that it might well be taken for a glass model, were it not for the cubic cleavage clearly visible on certain of the faces. Such a crystal may be considered as an extension of the simple magnesia lattice shown in fig. 59. This structure, which was first worked out by X-ray examination, is similar to that of common salt ( $NaCl$ ) except that the "black" atoms instead of being sodium are magnesium, and the "white" atoms are oxygen instead of chlorine. The charge on these atoms (or ions) is double that with sodium chloride and the electrical forces holding the structure together are therefore



*Fig. 59. The structure of magnesia. The two types of spot represent the two types of atom. It is immaterial which is taken to be the magnesium and which the oxygen.*

much stronger. This shows itself in a high refractoriness, melting only occurring when the thermal vibrations are sufficient to carry the ions beyond the control of these electrostatic forces. Incidentally the length of the unit cube edge is  $4.20 \text{ \AA}$  units.

It has been suggested from time to time that magnesia exists in a number of different forms, but it has now been shown by numerous workers that the variation in properties is merely due to differences in crystal size. All forms whether they be calcined at low temperature or high temperature, and whether produced by calcination of the carbonate or hydroxide, all have this simple cubic structure; the location of the lines in the Debye-Scherrer photographs being identical whether the material is lightly calcined or electrically fused. There is, however, as will be shown later (fig. 69, p. 136), considerable variation in line width. Even magnesium oxide smoke, formed when magnesium metal is burnt, is seen to be cubic when viewed at 50,000 magnification under an electron microscope. The fundamental structure of magnesia is of practical interest, since it enables an estimate to be made of the density ( $\rho$ ) of the pure material. It can be shown that this is given by the formula

$$\rho = \frac{4M}{d^3N}$$

where  $\rho$  is the density,  $M$  the molecular weight of magnesia,  $N$  Avogadro's number, and  $d$  the length of the unit cell edge. Using the value



*Fig. 58. Magnesia crystal  
( $1\frac{1}{2}$  times actual size) formed  
by prolonged heating at a  
high temperature.*





4.20 for the latter, a calculated density of 3.59 is obtained—a figure considerably below that quoted for Moissan by the International Critical Tables. In all probability the sample he used was impure, or the method insufficiently refined for use with this material. Following a series of apparently low results on electrically fused magnesia, the author attempted to make a particularly accurate determination using the single crystal shown in fig. 58, and the technique employed for determining the density of, say, a glass stopper. The value obtained as a result of weighing both dry, and immersed in water, was 3.583. The higher values obtained for example with Austrian magnesite may well be due to the iron oxide present, since the latter appears to raise the specific gravity by about 0.01 for each per cent. present.

#### MELTING POINT

The most commonly accepted value for the melting point of magnesia is that given by Kanolt, *viz.*, 2800°C. To the steelplant operator the precise figure is only of academic interest, since commercial magnesite bricks fail well below this temperature, due to the softening of the silicate bond. The fact that the ultimate melting point is, however, so much higher than that of other steelplant refractories augurs well for its future. Magnesia has, for example, been used for arch construction at temperatures as high as 2000°C.

#### REFRACTIVE INDEX

The refractive index of pure magnesia is given as 1.74. Like all other cubic minerals it is isotropic, *i.e.*, has uniform optical properties in all directions and, therefore, appears black when viewed on the petrological microscope in thin section under crossed nicols. Only occasionally do sections cut through magnesite bricks or grain show a marked cubic structure (*see*, for example, fig. 6, p. 19), more often they reveal a poikilitic or raspberry-like structure consisting of rounded grains held together by interstitial bond.

#### BINARY SYSTEMS CONTAINING MAGNESIA

##### *MgO—CaO*

This system (fig. 80, p. 169) is peculiarly important in that it shows that burnt dolomite of the type used for fettling does not (in the absence of other fluxes) form any liquid phase at temperatures below 2300°C. It is also of importance in that calcined dolomite ( $\text{CaO} + \text{MgO}$ ) is the raw material for the manufacture of semi-stable dolomite bricks and a

principal reactant in the sea-water magnesia process. It will be discussed in more detail in the next chapter.

### *MgO-FeO*

Magnesia forms a continuous series of solid solutions with ferrous oxide (*see* fig. 60) and here again a very large substitution of the refractory by the flux is necessary before a composition melting at open-hearth furnace temperatures is reached. It is therefore not surprising

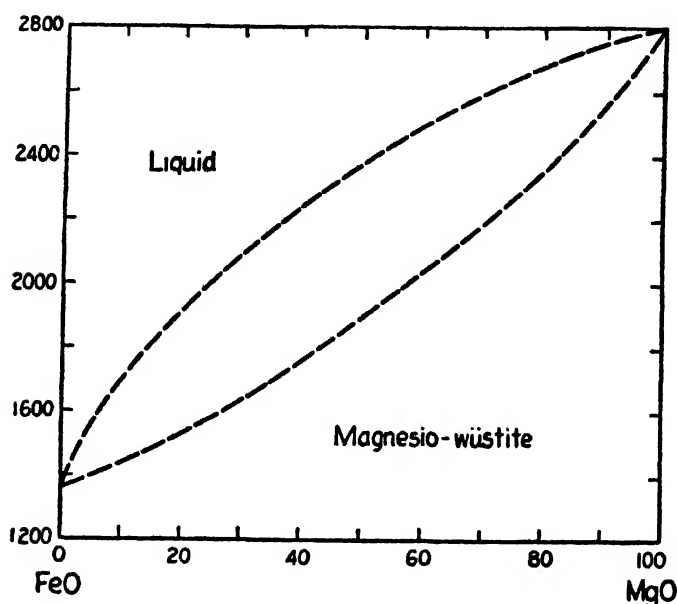


Fig. 60. System  $MgO-FeO$  (after N. L. Bowen and J. F. Schairer).

that the magnesite bricks and magnesite-chrome bricks used in open-hearth furnace roofs show very little signs of dripping, normally failing prematurely from other causes. It is for the same reason that magnesite bricks are sometimes used—very successfully—in the hearths of soaking pits containing liquid scale.

### *MgO-Al<sub>2</sub>O<sub>3</sub>*

*See* Chapter V and fig. 99 (page 204).

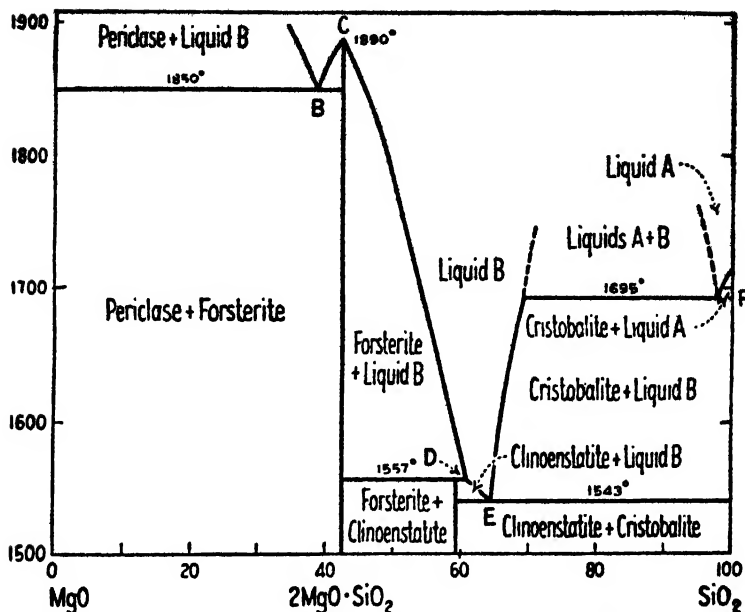


Fig. 61. System  $\text{MgO-SiO}_2$  (after N. L. Bowen and O. Andersen—modified by J. W. Greig).

### $\text{MgO-SiO}_2$

Fig. 61 is perhaps the most important of the binary systems containing magnesia. Unlike the two previously mentioned, it shows the presence of a eutectic having a melting point of only 1543°C., the composition of which is approximately 35 per cent. magnesia and 65 per cent. silica. Fortunately it also contains a compound—*forsterite* ( $2\text{MgO} \cdot \text{SiO}_2$ )—with a melting point of nearly 1900°C. Both natural and synthetic refractories of this composition are employed in steelplants and show a resistance to attack by iron oxide intermediate between magnesia and silica. The natural mineral, *olivine*, is usually a solid solution of forsterite with the corresponding iron mineral. Forsterite is also present in substantial amounts in high silica magnesite bricks and is the normal bond in chrome-magnesite refractories.

### TERNARY SYSTEMS CONTAINING MAGNESIA

#### $\text{MgO-CaO-Al}_2\text{O}_3$

Although refractories are not normally made from these oxides, the combination is liable to occur for example where magnesite is bonded

with ciment fondu (aluminous cement), or where dolomite is attacked by alumina rich slags. It has also been shown that a synthetic composition containing approximately equal amounts of lime and alumina and some 5 per cent. of magnesia has a very low melting point, and can be used on induction furnace casts without serious damage to the lining—even an increase in the magnesia content to 15 per cent. as a result of attack on the lining being sufficient to raise the melting point to over 1600°C.

#### *MgO-FeO-Fe<sub>2</sub>O<sub>3</sub>*

One of the reasons why Austrian magnesite was among the first to be used as a refractory was that the presence of iron in the natural breunnerite material mined in Austria greatly facilitated sintering and recrystallisation. The main importance of the system as distinct from that of the MgO-FeO binary system is that it indicates that the reaction product depends on the furnace atmosphere. Thus if the latter is strongly reducing the iron will remain in solid solution as FeO, whereas if it is oxidising magnesio-ferrite (MgO.Fe<sub>2</sub>O<sub>3</sub>) will tend to crystallise on cooling. The two states of oxidation are well illustrated by the pale green colour of electrically fused magnesia, and the chocolate brown colour of ordinary Austrian magnesite bricks. The system has been discussed very fully by Fisk and McCaughey.

#### *MgO-CaO-SiO<sub>2</sub>*

Since this system represents the scientific basis for the commercial production of stabilised dolomite bricks, detailed discussion of it will be left to Chapter IV. It is, however, also of importance in magnesia refractories, in that it shows that the ratio of lime: silica in the bond may determine whether relatively refractory minerals, such as dicalcium silicate (2CaO.SiO<sub>2</sub>), or less refractory compounds, like wollastonite (CaO.SiO<sub>2</sub>), will form in the clinker or brick. In certain high lime magnesites, e.g., Canadian, dicalcium silicate formation may be quite substantial and trouble experienced due to inversion to the  $\gamma$  form on cooling below 675°C. The dusting which is associated with this 10 per cent. volume change can be overcome by the use of stabilisers, such as borates and phosphates.

#### *MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>*

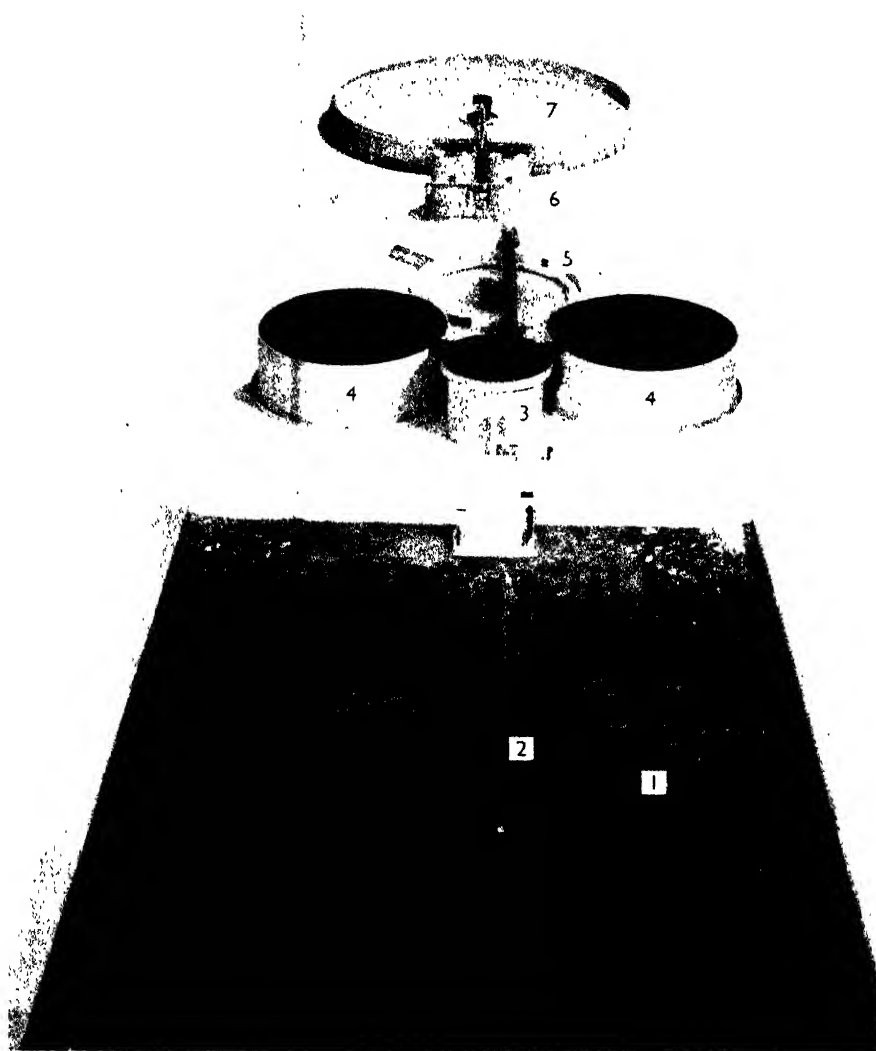
Alumina and silica normally occur in magnesite refractories to the extent of 1 or 2 per cent., but in at least one type of brick the alumina is added deliberately with a view to increasing its thermal shock resistance.



*Fig. 62. Austrian breunnerite (black) - dolomite mixture—prior to concentration. Approximately actual size.*

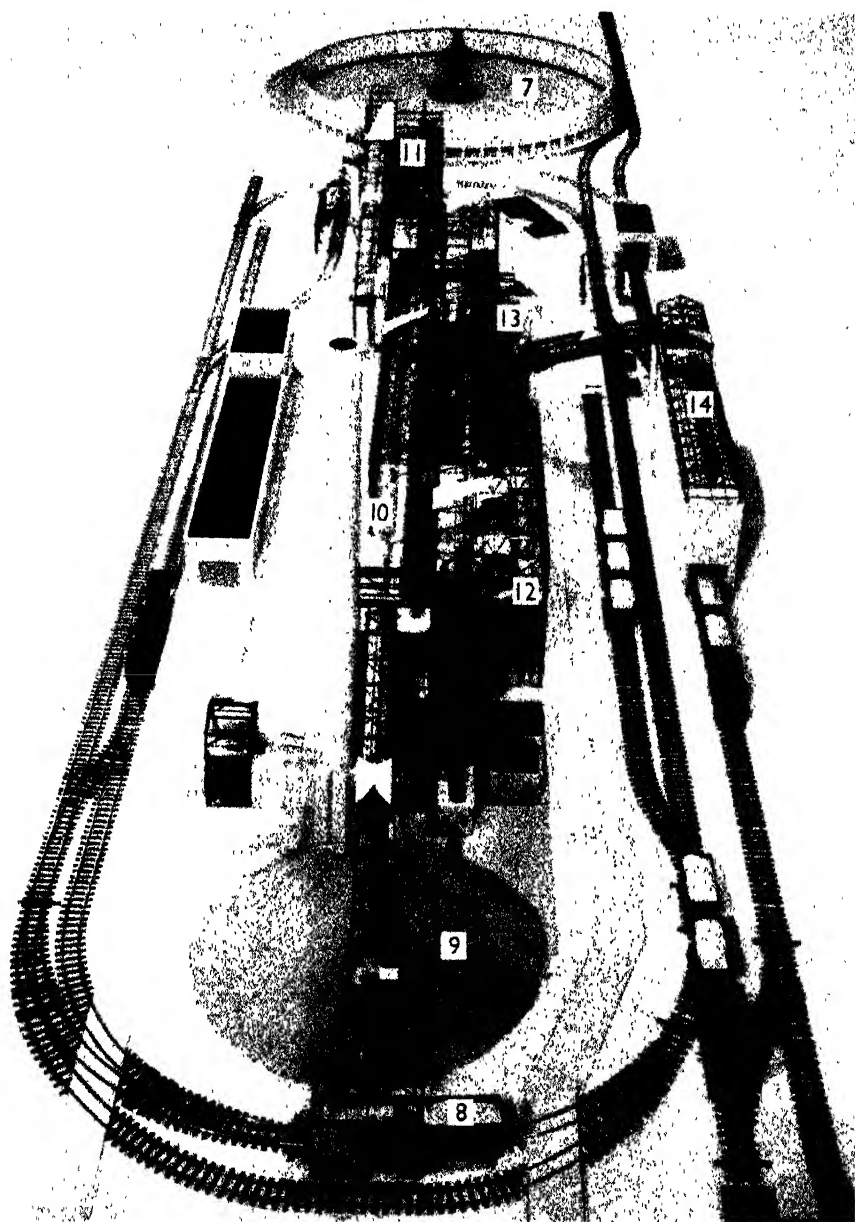


*Fig. 63. Grecian dead-burned magnesite, showing characteristic fibrous structure.*



*Fig. 64. Model of Palliser Sea Water Magnesia Plant.*

*1 Sea water; 2 pipe-line jetty; 3 sand trap; 4 sea-water storage tanks;  
5 water softener; 6 reaction tanks; 7 settling tank.*



*Fig. 64 (cont.). Model of Palliser Sea Water Magnesia Plant.*

*8 Dolomite stone; 9 stone stock-pile; 10 dolomite burning kiln; 11 slaking plant; 12 magnesia filters; 13 Britmag dead burning kiln; 14 Britmag storage bunkers.*





*Fig. 65. Dorr Thickener used for de-watering magnesium hydroxide suspension, at Palliser Magnesia Works, Hartlepool, Co. Durham.*

The eutectic in this system has a very low melting point, *viz.*,  $1345^{\circ}\text{C}.$ , but it would not be expected that this would be present in substantial amounts in a magnesia refractory fired at a high temperature. The fact that the eutectic ( $\text{MgO}$  20.3 per cent.,  $\text{Al}_2\text{O}_3$  18.3 per cent.,  $\text{SiO}_2$  61.4 per cent.) can be formed by reaction between magnesia and a firebrick indicates the desirability of separating these refractories by a suitable neutral material, or alternatively ensuring that they are not used in contact above, say,  $1300^{\circ}\text{C}.$

#### *MgO-FeO-SiO<sub>2</sub>*

This system has already been mentioned in Chapter II, figs. 43 and 44, from which it can be seen that whereas  $\text{FeO}$  and  $\text{SiO}_2$  form low melting point mixtures ( $1177^{\circ}\text{C}.$ ) the addition of only 20 per cent. of magnesia brings the melting point above  $1600^{\circ}\text{C}.$

### QUATERNARY SYSTEMS CONTAINING MAGNESIA

#### *MgO-CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>*

This system which was recently worked out in some detail by Osborn, Devrics, Gee and Kraner, has proved invaluable in connection with blast furnace operation and will doubtless also prove of interest to those concerned with the composition of the bond in magnesite bricks. It also provides valuable information regarding possible reactions between fettling dolomite and fireclay brick.

The quaternary system ( $\text{MgO-Al}_2\text{O}_3\text{-CaO-Fe}_2\text{O}_3\text{-SiO}_2$ ) has not yet been worked out, and even when it has been will present quite a problem in presentation of data. The above mentioned paper by Osborn and co-workers should serve as a model of how such information can be communicated to those relatively unfamiliar with equilibrium diagrams.

## RAW MATERIALS

### (a) SOURCES

In spite of the remarkable developments already referred to in the field of sea-water magnesia, the natural mineral is still the principal source of raw material. Incidentally this competition raises the question as to whether it is permissible to use the term "dead-burned magnesite" for material which never has been magnesite, *i.e.*, the carbonate, at any time in its history. The term "dead-burned magnesia" would seem preferable.

### 1. Coarsely crystalline magnesite:

The first refractory magnesite appears to have been mined at Veitsch, Austria, in about 1880. This country still supplies a very substantial part of the world's requirements but Russian and Manchurian outputs are also large. The Austrian product is frequently referred to as breunnerite, since it is a solid solution of magnesium and iron carbonates, the amount of iron being generally such as to yield 4 to 8 per cent. of  $\text{Fe}_2\text{O}_3$  in the dead-burned product. Coarsely crystalline magnesia also occurs in the State of Washington and in Czechoslovakia. Most breunnerites as mined contain substantial amounts of dolomitic impurity (*see* fig. 62, p. 127) and are, therefore, concentrated either by crushing, screening and washing before calcination, or by magnetic methods after firing in vertical shaft kilns.

### 2. Crypto-crystalline or "compact" magnesite:

This type of magnesite, which is normally white in colour and very hard, is found in large pockets in Greece—particularly in the island of Euboea—and also in Yugoslavia, India, Australia, South Africa, and California. It is normally low in iron and is peculiarly suitable after light calcination for the production of Sorel (oxy-chloride) cement or magnesium metal. If fired at a sufficiently high temperature (*see* fig. 63, p. 127) provides an excellent raw material for refractory brick manufacture.

### 3. Brucite:

This naturally occurring form of magnesium hydroxide is also found in considerable quantities in Canada (Ontario and Quebec) and to a less extent in Nevada. Generally it is associated with substantial amounts of other minerals and must be greatly concentrated before being suitable for high grade refractory work.

### 4. Sea-water magnesia:

Virtually the whole of the refractory magnesia used in Great Britain now comes from the sea-water process, whilst nearly half of that in America is similarly produced. The outputs from sea-water magnesia plants during the war were even higher, but the bulk of the product was used for magnesium metal production.

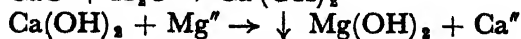
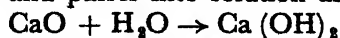
## (b) PRODUCTION OF SEA-WATER MAGNESIA

The extraction of magnesia from sea-water is only made economic by the extremely low solubility of magnesium hydroxide, which enables it to be precipitated from solutions of its salts by bases capable of providing a moderate concentration of hydroxyl ions. The first step in the very simple chemistry of the dolomite—sea-water process, is the

calcination of the dolomite to give a mixture of calcium and magnesium oxides:



When the product is added to sea-water, the calcium oxide after hydration reacts with the magnesium salts, precipitates magnesium hydroxide, and passes into solution as follows:



The magnesium oxide undergoes some hydration, but is otherwise unchanged and is recovered with the precipitant.

In actual practice the process, shown diagrammatically in fig. 64, pages 128 and 129 is considerably more complicated than these equations suggest, since:

- (1) Insoluble impurities in the calcined dolomite (dolime) are recovered with the magnesium hydroxide.
- (2) The addition of lime to sea-water results in a precipitation of other compounds (mainly of calcium) in addition to magnesium hydroxide.
- (3) The magnesium content of sea-water expressed as MgO is only 0.2 per cent., and 300 tons of sea-water must be pumped in order to get 1 ton of product.

The principal insoluble impurities in dolime are the iron oxide, silica and alumina derived from the dolomite, calcium carbonate arising from incomplete calcination and fuel ash. To reduce these to acceptable levels generally necessitates some measure of mineral dressing, even when the highest quality raw materials are employed, and great care with the control of the calcination process. Washing and heavy media separation have been successfully applied to the raw dolomite, particularly in the United States, whilst air classification has been used in Great Britain, and hydraulic classification to the hydrated dolime both in Britain and the United States.

The chief impurity coming from the sea-water is calcium carbonate, due to carbon dioxide being held in solution mainly as bicarbonate. Such contamination can be reduced to a minimum by treating the water prior to precipitating the magnesium hydroxide. This treatment is similar to that employed for removing the bicarbonate hardness from fresh water, and is therefore commonly referred to as "softening". Dorr Hydrotreators are mainly employed for this purpose both in British and American plants. An alternative method is acidification of the sea-water to a pH of 4 followed by aeration to blow out the carbon dioxide. This method is, however, expensive unless waste acid can be employed.

Another potential contaminant from the sea-water is calcium sulphate. Fortunately this forms relatively stable super saturated solutions and its precipitation can therefore be avoided by careful control of the process. Sea-water magnesia also contains certain minor impurities from the sea-water, one of the more interesting being boron.

The next problem, that of separating the magnesium hydroxide from the vast quantities of water, was one which had to be tackled very vigorously before there was any chance of this process becoming a commercial proposition. A study of the patent literature shows that numerous ideas have been tried out but it now seems generally recognised that the maximum rate of settling and ease of de-watering are obtained by so controlling reaction conditions as to promote the formation and growth of crystalline hydroxide. Two of the main techniques involved are the use of some form of a seeding technique and the control of  $pH$  (acidity) in the reaction mixture.

After precipitation the hydroxide is continuously settled in Dorr Thickeners and the sludge from these further concentrated by means of disc or drum type rotary vacuum filters. A cake having a hydroxide content of 50 to 60 per cent. is obtained. This is then fed to rotary kilns lined with basic brick where it is dead-burned using either oil or pulverised coal as the fuel. During such calcination both the free and combined water are driven off, together with the salts of alkali metals, whilst any carbonates or sulphates present are decomposed.

#### Hartlepool Plant:

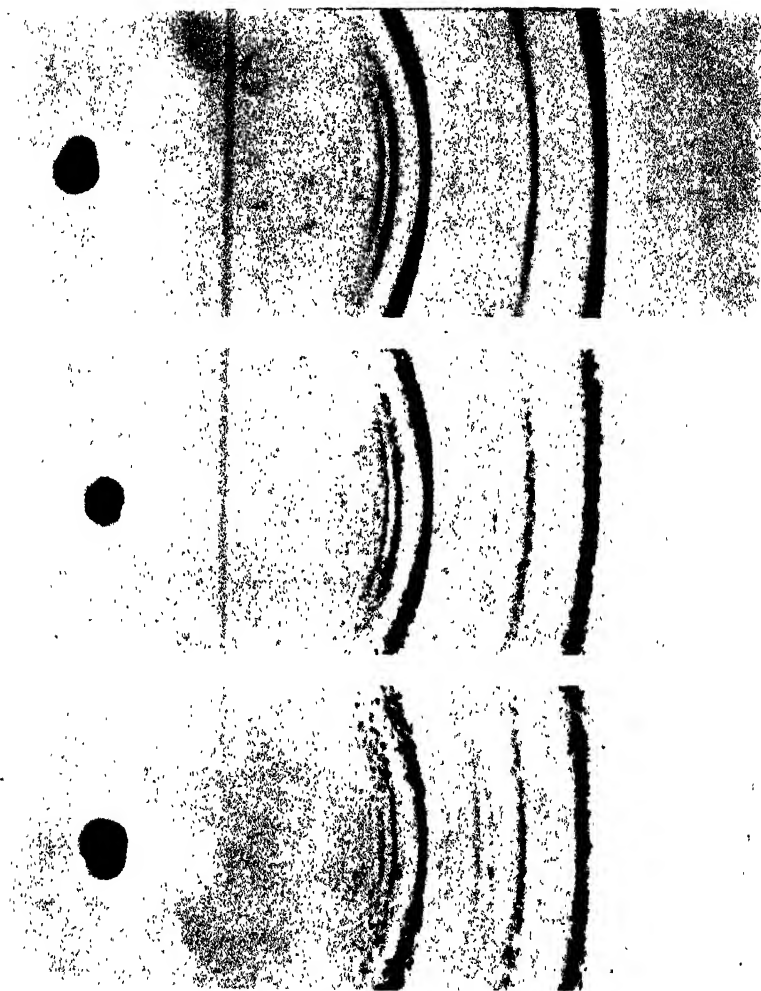
The first plant to be built for the production of sea-water magnesia for refractories, using calcined dolomite for the reactant, was that at Hartlepool, County Durham, now known as the Steetley Magnesite Company Limited—a subsidiary of the Steetley Company. Certain quantitative information regarding this plant should serve to illustrate the information given above.

The Permian dolomite which comes from the Coxhoe quarry, some 15 miles from Hartlepool, is crushed into a range of sizes, two of which—the 6 in. to 4 in. and the  $1\frac{1}{2}$  in. to  $\frac{1}{2}$  in.—are calcined in vertical and rotary kilns respectively. The shaft kilns are 68 ft. high and 11 ft. internal diameter. They are coke fired and have a thermal efficiency of 80 per cent. The product from these shows less than 1 per cent. ignition loss compared with a figure of less than 0.3 per cent. for the rotary kiln product. Both types of material are then crushed to less than  $\frac{1}{2}$  in. by means of rolls and are transported to Hartlepool.

Here the dolime is hydrated in Sturtevant Hydrotreaters, only sufficient water being added to give a free flowing powder, that can be



*Fig. 66. Filtration of magnesium hydroxide slurry—sea-water magnesia process, at Palliser Works, Hartlepool, Co. Durham.*



*Fig. 69. X-ray side reflection photographs of Indian magnesite after firing at: top—1300°C.; middle—1600°C.; bottom—1650°C.; showing increasing crystal size.*

air classified to remove impurities introduced from the fuel during calcination, and from the dolomite. This material is then mixed to a slurry with sea-water to facilitate its transfer to the reaction vessels.

The sea-water is pumped through four 22 in. diameter pipe lines 900 ft. in length to two reinforced concrete tanks, each having a capacity of 2 million gallons and being 106 ft. in diameter and 40 ft. in depth. From here it is taken to the Dorr Hydrotreaters, where a small quantity of hydrated lime slurry is used to precipitate the bicarbonate ion as calcium carbonate. This treated sea-water overflows into a small reservoir tank and is then pumped to mechanically agitated reaction tanks, where it is intimately mixed with hydrated dolime slurry. The proportioning of the dolime with this sea-water must be carefully controlled by chemical tests in order to ensure that at least 95 per cent. of the magnesium ions are precipitated. From here the suspension passes to the Dorr Thickeners, where the magnesium hydroxide settles to give a thick sludge. These huge concrete tanks (*see* fig. 65, p. 130) are 240 ft. in diameter, and have a depth of 22 ft. at the centre. They are fitted with continuously operated centrally driven rake mechanisms. The sludge from these primary thickeners must then be washed with a small quantity of sea-water to adjust lime impurities and then be thickened once again. The final stage of slurry concentration makes use of rotary vacuum disc filters (*see* fig. 66, p. 135), these are 9 ft. 6 in. in diameter, the latest filter medium being nylon. The product from these, which contains 55 per cent. of magnesium hydroxide, is conveyed to the pulverised coal fired rotary kilns, where it is burned at a temperature of 1600° to 1650°C.

#### (c) PRODUCTION OF DEAD-BURNED MAGNESITE

By "dead-burned" is meant a magnesite that is unreactive, *i.e.*, "dead" because it has been calcined at a temperature high enough to enable it to be used in brick-making or monolithic hearths without undue difficulty due to hydration or shrinkage. Whether the material is made from the carbonate or the hydroxide the same general considerations apply, though the precise reactions are, of course, different. The dissociation of magnesium carbonate was studied by many workers, even before the close of the last century. It has since been studied in detail by Conley in connection with calcination conditions for limestone, dolomite and magnesite, who showed that dissociation is very rapid at about 600°C. (fig. 67). This conclusion was also reached by the author, who showed that the breakdown temperature of the carbonate is considerably higher than that of the hydroxide, while the basic carbonates dissociate over a long and intermediate temperature range.



The main point of interest to the steelplant operator is the effect of the calcination temperature and the fluxes present on the rate of crystal growth and the stability of the final product. The data summarised in

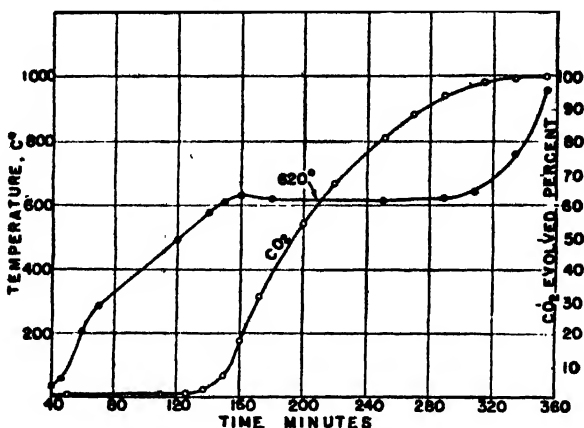


Fig. 67. Dissociation of magnesium carbonate (after J. E. Conley).

Table VIII and fig. 68 give an example of the kind of result obtained. These particular tests happen to have been made on a sample of lightly calcined Indian magnesite, having the following compositions:

MgO	..	..	..	90.8 per cent.
CaO	..	..	..	2.6 „
SiO <sub>2</sub>	..	..	..	5.4 „
Fe <sub>2</sub> O <sub>3</sub>	..	..	..	0.7 „
Al <sub>2</sub> O <sub>3</sub>	..	..	..	0.4 „

The material was ground to pass a 72 mesh (B.S.I.) sieve, moulded into cylinders and fired in a laboratory gas-fired kiln. Above 1000°C. the heating rate was kept steady at 200° per hour to 1650°C., this being the order of heat treatment received in a rotary kiln. Samples were withdrawn every half-hour and specific gravity tests made. It will be seen from fig. 68 that above 1400°C. the specific gravity began to rise quite rapidly, reaching a maximum value of 3.56 at 1650°C. The crystal size, as determined by the X-ray monochromatic pinhole method, increased from 0.01 mm. to 0.05 mm. The change in crystal size is clearly shown by the X-ray photograph (fig. 69, p. 136). The hydration tendency, which was determined by maintaining a small sample in contact with steam for 5 hours, also fell off rapidly with increased firing temperature, being less than one-quarter at 1500°C. of

TABLE VIII

INFLUENCE OF FIRING TREATMENT ON THE SPECIFIC GRAVITY, CRYSTAL SIZE AND HYDRATION TENDENCY OF COMPACT MAGNESITE

<i>Treatment (rising temperature)</i>	<i>Specific gravity</i>	<i>Range of crystal size</i>	<i>Hydration tendency— five hours in steam— loss on ignition</i>
		<i>diam. (mm.)</i>	<i>per cent.</i>
Sampled at 1300°C. ..	3.494	0.01–0.03	8.91
" " 1400°C. ..	3.496	0.01–0.02	7.36
" " 1500°C. ..	3.539	0.02–0.03	1.92
" " 1600°C. ..	3.544	0.03–0.04	1.13
" " 1650°C. ..	3.551	0.04–0.05	1.06
Held half hour at 1650°C.	3.565	0.04–0.05	1.04

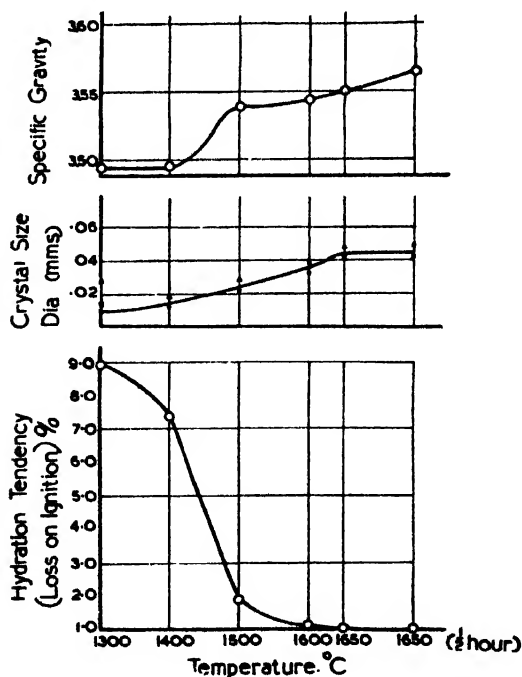


Fig. 68. Effect of firing treatment on the specific gravity, crystal size and hydration tendency of lightly calcined compact magnesite.

the value obtained for the 1300°C. sample. These conclusions illustrate the general problem which is analogous for all the materials involved: on firing the magnesia crystals grow, the amount of surface exposed is reduced and there is a drop in the hydration tendency. As the material shrinks as the minute pores in the magnesia disappear and the specific gravity increases.

A great deal of work has been done on the effect of various fluxes on the rate at which these changes occur and numerous patents have been taken out to cover the materials used. Thus, in the Alterra process, calcium ferrites are added to pure magnesite of the compact type to increase the rate of dead-burning and reduce the risk of after-contraction in the final product.

Those who wish to study the types of periodic and rotary kiln employed in the dead-burning of magnesite, are referred to the excellent books by Comber and Banco referred to in the bibliography. A description of the Austrian plants has also been given in some detail by Endell. At the Radenthein works, where a breunnerite yielding magnesia with 4 per cent.  $\text{Fe}_2\text{O}_3$  content is dead-burned, large rotary kilns capable of producing 200 tons of magnesite per day are employed. The material is fed in as lumps of about 1 inch size and is calcined at a temperature between 1650° and 1750°C. The advantage of the rotary kiln over the earlier periodic shaft kiln type is, of course, the uniformity of the firing treatment.

Mention should also be made of electrically fused magnesia, which although at present only a small proportion of the total, will doubtless be used in increasing amounts as the demands on furnace structures increase. It has been found to give quite satisfactory results in ports and at the slag line in basic open-hearth furnaces where the conditions are particularly severe. The extended use of this material is mainly a question of the cost of the electrical power required for fusion.

#### (d) PROPERTIES OF DEAD-BURNED MAGNESITE GRAIN

##### *Chemical composition*

Selected chemical analyses of the principal brands of magnesite are given in Table IX. In general the Veitsch magnesite runs about 8 per cent. iron oxide and the Radenthein and Trieben about 4 per cent. iron oxide. The silica content varies from 2 to 4 per cent. and the alumina is generally about 1 to 2 per cent. The lime is usually less than 3 per cent. This analysis is not obtained automatically, but results from careful sorting of the raw materials and even magnetic separation of the fired product.

The Greek magnesite is generally low in iron whilst the magnesia usually exceeds 90 per cent. Most of it comes from deposits in the island of Euboea, though considerable amounts also occur on the mainland and in the island of Mytilene. It is a difficult material to dead-burn, though complete success has been achieved by the Anglo-Greek Magnesite Co. (*see* fig. 63, page 127). The difficulty lies in the fact that a very high temperature is required and that, in the shaft kilns used, caustic material can exist side by side with dead-burned magnesite. The use of mineralisers in shaft kilns has not been successful, and consequently very careful supervision and sorting of the product is required to ensure that it is up to standard.

Russian magnesite is similar in composition to Austrian, but tends to be lower in iron oxide. A possible difficulty with its use may be the presence in the clinker of a considerable amount of free lime. If this occurs as discrete particles of appreciable size it hydrates during the drying of the brick and causes cracking. A great deal of work has been done in Russia on the pre-treatment of the clinker to eliminate the more active lime and magnesia. One method that has been tried is the autoclaving of the dead-burned magnesite, using a plant similar to that employed in the production of sand-lime brick. The advantage of this method is its rapidity and the consequent large tonnages that can be handled by a comparatively small plant. Experiments made in Great Britain suggest that autoclaving may easily be excessive and point to souring the brick batch on a hot floor as a safer method.

Manchurian magnesite was extensively used both in Great Britain and in America prior to the war and offers no particular problem as a brickmaking material. The Canadian magnesite is generally high in lime, a figure of 18 to 20 per cent. being typical. Stability is achieved by the addition of iron oxide in the rotary kiln, the amount of iron present generally being about 8 per cent. With all these fluxes present the material can scarcely be considered as straight magnesite. Nevertheless, it has found useful applications, both in brickmaking (magnesite and chrome-magnesite) and in the production of a variety of stabilised clinkers and cements. Washington magnesite is also quite pure, but in brickmaking behaves rather differently from Austrian magnesite, due to its relatively low content of iron oxide. It is extensively used in the States, both in brick form and as a clinker for open-hearth furnace bottoms.

Sea-water magnesia, an analysis of which is given in Table IX, can vary considerably in composition according to how the process is worked. Thus for pharmaceutical purposes an extremely pure product is obtained, but for refractory work a content of about 90 per cent. of

TABLE IX

SELECTED ANALYSES OF DEAD-BURNED MAGNESITE

Source	Austria		Greece	Russia	Man- churia	India	Canada	United States	Sea- water mag- nesia
	Veitsch	Radenthein	Euboea					Wash- ington	
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
Silica ..	1.7	2.1	2.6	4.7	2.8	5.4	7.9	3.2	2.5
Alumina	0.9	2.4	0.4	1.1	2.3	0.4	0.2	3.2	1.5
Ferric Oxide	8.2	3.9	0.5	2.7	1.9	0.7	7.8		2.0
Lime ..	2.5	2.3	2.0	5.7	1.8	2.6	19.5	2.0	2.5
Magnesia	86.9	89.0	95.3	85.2	90.8	90.8	64.5	91.6	91.5

magnesia is normal. Silica, iron oxide and alumina, are all present to the extent of about 2 per cent., while the lime content varies from about 3 to 6 per cent. It is generally agreed that the lime should be as low as possible, and a value of  $2\frac{1}{2}$  per cent. is now being achieved by close control over the raw materials and processes.

#### *Specific gravity, crystal size, hydration tendency and firing shrinkage*

Attempts have been made in the past to assess the value of a magnesite clinker from a single test. Experience gained during the last few years shows how exceedingly dangerous such a procedure can be. It is now considered that at least 6 tests should be done, namely, chemical analysis, specific gravity, crystal size, bulk density, hydration tendency and firing shrinkage. Both the bulk density and the specific gravity afford a rough index of the degree of dead-burning, but must always be considered in relation to the chemical analysis. Thus the Radenthein magnesite, which contains 4 per cent. of iron oxide, generally has a specific gravity of 3.56, while that of Veitsch which contains 8 per cent. of iron oxide, may be as high as 3.65. For the very pure Greek material a value of 3.50 may be considered quite good. The specific gravity figures may be corrected to the extent of 0.01 for every 1 per cent. of iron oxide, though this is only a rough approximation, as it takes no account of other impurities, *e.g.*, silica. The crystal size supplies confirmatory evidence of the degree to which the material is dead-burned—well burned material being usually 0.03 mm. or over—but from the brickmaking standpoint the two important characteristics are the

hydration tendency and the firing shrinkage. If the hydration tendency exceeds the safe value or if the material contains a small amount of highly reactive material, then trouble may be anticipated in drying the bricks. In view of this latter qualification the hydration figure alone may be misleading, and it may be necessary to pick out discrete particles of material for separate test. Firing shrinkage tests are again only comparative, but if the firing shrinkage of a new material is considerably greater than normal, cracking is likely to occur in the kiln and the brick, if made in standard moulds, will be under size.

#### *Sintering tendency*

The heat-treatment required to sinter magnesite is a factor of some importance, particularly in connection with monolithic hearths. The finer the grind and the less pure the material, the more easily will sintering occur. Needless to say, an increase in temperature or time of soak increases the amount of sintering.

### MANUFACTURE OF MAGNESITE BRICKS

For many purposes a hard-fired magnesite brick of the type first made by the Veitsch Company since 1880 is still preferred, though such bricks are now made from a wide variety of raw materials, including sea-water magnesia. The most remarkable development over the last 20 years has been the substantial replacement of fired bricks by the unfired type, which are normally made with a steel jacket or used in conjunction with loose steel plates. The idea of a steel case is far from new, having been employed for example by McCallum as far back as 1920, but it was not until it was demonstrated, for example, by Fitzgerald, that chemically bonded bricks were capable of being produced that suffered less in handling than fired bricks and gave equally good service in open-hearth furnaces that the present changeover began.

The advantages of the unfired brick apart from a somewhat lower initial cost are speed of delivery, accuracy of shape, and toughness in handling. In the early days such bricks were used with loose steelplates, but these have now been largely superseded by plates attached to the brick surface. Thus in one procedure a "U" shaped plate is forced into the press box before the magnesia is fed into it and a plate attached to the top plunger and fitted with lugs completes the armouring of the 4 largest sides of the brick. Bricks of this type are extremely strong and can readily be suspended either by means of a built-in clip or by welding to the steel case.

## GRINDING AND GRADING

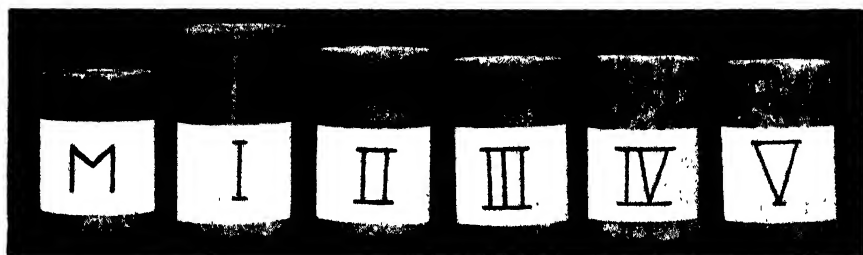
In the early days of brick manufacture pan mill grinding of relatively wet batches was frequently practised, but today the normal procedure is to grind the dead-burned peas and to sieve into 2 or 3 size sections prior to recombination in a mixer having raised mullers to avoid any further grinding. The importance of grading is illustrated by the test cylinders shown in fig. 70, opposite, the only differences between those marked I to V being the grading employed. The unfired test pieces varied in porosity from 37 per cent. for No. I to 24.5 and 22.5 per cent. for Nos. IV and V, made respectively from 70-0-30 and 70-10-20 coarse-medium-fine type batches. The grading of a magnesite brick also has a marked effect on the firing shrinkage, which is usually greatest on bricks having high unfired porosity or a small average grain size. For most purposes where magnesite bricks are specified, *e.g.*, open-hearth and arc furnace bottoms, or inactive mixers, extremely dense bricks are desired, though there may be positions, *e.g.*, arc furnace roofs, where a magnesite brick behaves better if it has a relatively porous structure. Alternatively the porosity for such purposes may be normal but the average grain size very high, a procedure illustrated on the Continent by the war-time development of coarse grain magnesite bricks for use instead of chrome-magnesite in all basic furnace roofs.

## ADDITIONS

With many types of magnesite grain a satisfactory brick can be produced from a graded clinker merely with a small addition of water. For special purposes, *e.g.*, where great density is required, additional iron oxide may be added, whilst for increased thermal shock the use of 6 per cent. alumina has been specified. Advantageous results are also claimed for the use of soluble chromates yielding magnesium chromite in the fired product. Larger additions, *e.g.*, substantial quantities of either chrome ore or silica must be considered as outside the normal field of magnesite bricks.

## SOURING

Until comparatively recently, souring, *e.g.*, the mixing of clinker or brick batch with water and subsequent standing, was considered one of the secrets necessary to the success in magnesite brick manufacture. It is now recognised, however, that given a magnesite low in free lime or other caustic material, simple grinding followed by water addition (approximately 5 per cent.) and subsequent pressing is quite satisfactory. When, however, a magnesia contains a proportion of soft-fired



*Fig. 70. Magnesia test-pieces, showing the influence of grading on the bulk density. All the test-pieces were of the same weight and were moulded under similar pressure, except M, which was pressed at 56,000 instead of 21,000 lb. per sq. in.*





*Fig. 72. Magnesite block cracked due to excessive hydration in the drier.*

material or a serious content of free lime, such as may occur when the raw material is dolomitic, it may necessitate souring in some form. The choice is normally between allowing wetted clinker or brick batch to stand either on a cold or a hot floor and the treatment of the material by autoclaving with steam in closed drums. Where such practice is employed care must be taken to avoid over-hydration.

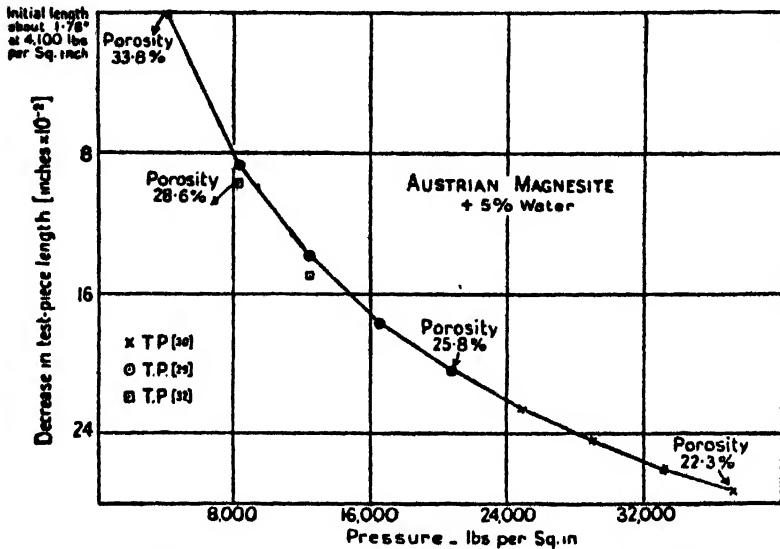


Fig. 71. Effect of moulding pressure on the porosity of magnesia test-pieces.

## MOULDING

Whether this be done by hydraulic, mechanical or hammer type presses, loads equivalent to approximately 10,000 lb. per sq. in. are now considered necessary. This results in a much denser brick than was normally obtainable by hand moulding or light mechanical pressure. That such hard pressing does result in improved porosity is shown by fig. 71 and Table X, where the effect of increasing moulding pressure from approximately 4000 lb. to 40,000 lb. per sq. in. is illustrated. Incidentally the table also shows that the harder pressed bricks show a somewhat lower firing shrinkage.

## DRYING

Even as late as 1931 the cracking of magnesite bricks in driers was considered to be something of a mystery. Work carried out by Weyl and the author showed that such cracking was normally due to:

**TABLE X**  
**INFLUENCE OF MOULDING PRESSURE ON SIZE AND POROSITY**  
**OF AUSTRIAN MAGNESITE TEST PIECES**

<i>Applied pressure</i>	<i>Decrease in test piece length (thousandths of an inch) above 4100 lb. per sq. in.</i>			
	T.P. (28)	T.P. (32)	T.P. (29)	T.P. (30)
<i>lb. per sq. in.</i>				
4100	0	0	0	0
8300	—	98	86	87
12,400	—	150	138	139
16,600	—	—	177	177
20,700	—	—	205	204
24,800	—	—	—	226
29,000	—	—	—	244
33,100	—	—	—	261
37,200	—	—	—	273
Final length (unfired)	1.783"	1.653"	1.604"	1.522"
Porosity unfired ..	33.8%	28.6%	25.8%	22.3%
Porosity after two hours at 1450°C. ..	26.1%	21.8%	18.8%	15.4%
Firing shrinkage—				
Vertical .. ..	6.3%	4.0%	3.9%	3.8%
Horizontal .. ..	2.2%	2.3%	2.2%	2.5%

(1) The use of soft-fired magnesite.

(2) The presence in the brick of excessive fines having a hydration rate considerably greater than coarse material.

(3) Too high a temperature in the drier.

At that time it was not uncommon for bricks containing a lot of fine material to be slop moulded and then dried at temperatures as high as 100°C., whereas this research showed that a rise in temperature even from 40° to 60°C. might result in considerable growth in drying and consequent cracking. The problem was particularly severe with large shapes since the drying time is longer and the inside dries at a different rate to the outside. The effects of this are illustrated by fig. 72, p. 146.

At a later date Chesters and Howie made continuous measurements on hydration expansion, obtaining curves of the type shown in fig. 73. It will be seen from these that the presence of calcined dolomite or caustic magnesia have a very marked effect on the expansion obtained during drying. Ford and Rees reported in 1948 continuous apparatus

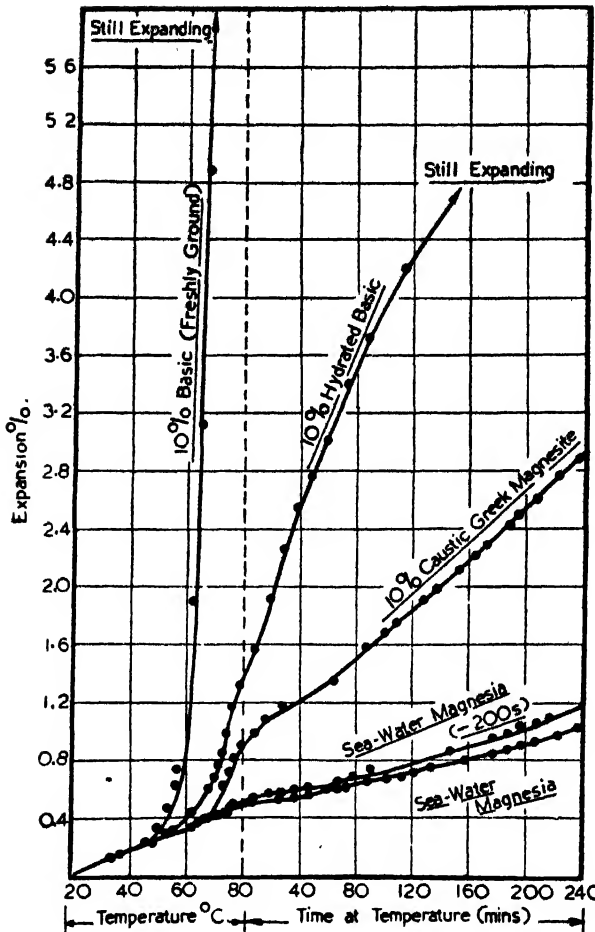


Fig. 73. Influence of various additions on the hydration-expansion of magnesia at 80°C.

capable of even more precise results, a sketch of which is shown diagrammatically in fig. 74. Incidentally Schreiner using a radioactive tracer technique showed that hydration began at the reactive centres in the brick and proceeded along the boundary surface between the periclase crystals and the bond. His work indicates that if 2 per cent. of the water reacts to form magnesium hydroxide the brick is liable to crack. That hydration troubles are not limited to brickworks was well illustrated in our own Company by failure of high grade, and otherwise satisfactory, magnesite bricks in the hearth of a soaking pit, due it is thought to the slow heating-up of material set with very wet cement.

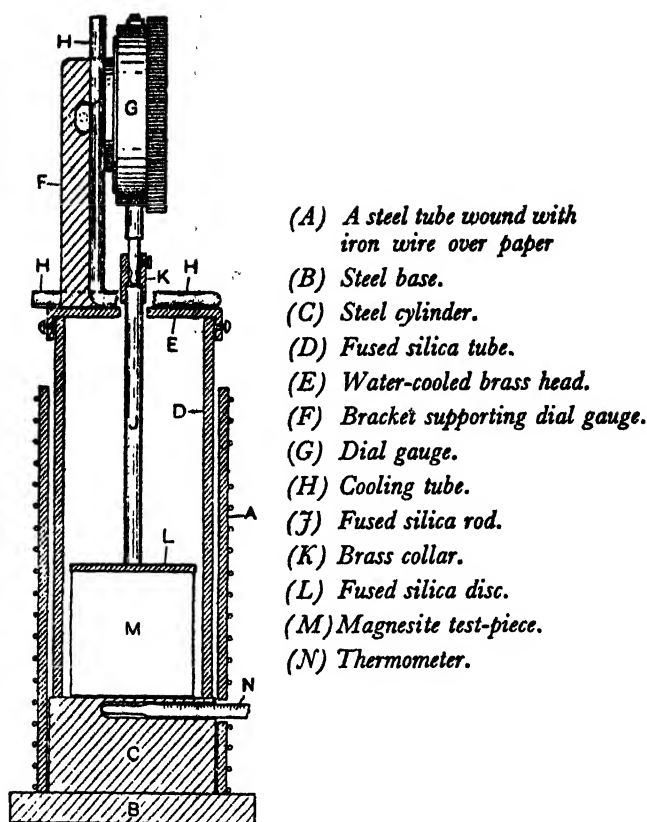


Fig. 74. Diagrammatic representation of hydration expansion apparatus (after Ford and Rees).

## FIRING

The first indication of the hydration problem arose from an investigation of the cause of cracking of special shapes in a brick kiln. Bricks which had not cracked at all visibly disintegrated completely when heated-up on a normal firing schedule. With modern methods of manufacture, where the small amount of moisture is removed in tunnel driers or on heated floors, no such difficulty is experienced, the problem being mainly to fire to a high enough temperature to avoid serious after-shrinkage without getting excessive movement or slumping in the setting. The use of tunnel kilns has greatly simplified the problem even though it does result in some inflexibility. With such kilns the relatively

low stacking height makes high temperature firing more practical than in periodic kilns, though extremely hard firing can be done in this latter type, as has been well illustrated by certain Austrian firms.

## THE PROPERTIES OF MAGNESITE BRICKS

The properties of the standard type magnesite brick as used, for example, in the sub-hearths of electric arc and open-hearth furnaces vary considerably but may be considered as represented by the selected bricks labelled Code No. M.4, M.17 and M.24 in Table XI. It will be seen from this that the typical porosity is of the order of 20 per cent. whilst the specific gravity varies from 3.52 upwards according to the iron oxide content. Bricks with as much as 8 per cent. of iron oxide present may have a specific gravity as high as 3.65. The cold crushing strength is normally very high over, say, 5000 lb. per sq. in., but the refractoriness under load relatively poor compared with either silica or chrome-magnesite bricks. The after-contraction should be low (1 per cent. or less after 2 hours at 1500°C.)—a specification not difficult to obtain in a brick fired at a sufficiently high temperature. The thermal shock resistance is very much a matter of grading and with straight magnesite brick is not generally considered particularly important. It is interesting to note, however, that sea-water magnesia bricks made with a 45-10-45 type grading and fired at a medium temperature show a thermal shock resistance of at least 30 reversals in the small prism test.

Numerous attempts have been made to produce bricks consisting substantially of magnesia but prepared either with additions or special heat treatment to make them suitable for specific uses. Thus the electrically sintered Grecian magnesite brick, Code M.1, was developed for positions in which thermal shock was important and where reasonably high refractoriness under load was required. It was, for example, found capable of giving service at least equal to chrome-magnesite bricks in the port nose of producer gas-fired furnaces. Brick M.2, which contains a substantial addition of alumina, was originally designed for use in open-hearth roofs, but has found its greatest value in rotary kiln linings. Electrically fused magnesia bricks, such as M.3, possess excellent properties under clean conditions, but tend to show the same structural spalling as standard magnesite bricks when used, for example, in back-walls. Such bricks are, however, now employed, particularly on the Continent, at the slag line in open-hearth furnaces. The unfired chemically bonded magnesite brick (M.5) is one of a large family of bricks now available, whose properties are particularly difficult to specify, in that the brick being unfired changes its character immediately on going into service. Even the low porosity (13.2 per cent.) may

TABLE XI  
PROPERTIES OF MAGNESITE BRICKS

Code No.	M4	M17	M24	M1	M2	M3	M5
Type							
Apparent porosity, per cent.	24.2	18.7	19.7	22.2	18.5	21.4	13.2
Bulk density—g.p.m.l.	2.67	2.89	2.83	2.76	2.90	2.80	2.94
lb. per cu. ft. . .	167	181	177	173	181	175	184
Apparent solid density g.p.m.l.	3.52	3.56	3.53	3.54	3.56	3.56	3.39
Cold crushing strength—on end-lb. per sq. in. . .	7170	>8300	—	1470	1860	—	5120
Permeability to air—perp. 9 in. X 3 in. face—1 skin. c.g.s. units	0.08	0.12	0.02	0.088	0.039	0.073	0.0044
Permanent linear change on reheating, 2 hrs. 1500°C. . .	1.0%	0.1%	—0.25%	—0.3%	—0.3%	—0.0%	+0.3% (Exp.)
Refractoriness—under-load—maintained temp. test—25 lb. per sq. in. @ 1600°C.	Failed in 11 mins.	—	—	—	—	Failed after 20 mins.	Failed after 12 mins.
Rising temp. test—	—	—	—	—	—	—	—
Initial softening . .	—	1450°C.	1490°C.	1480°C.	1410°C.	—	—
Rapid softening . .	—	1520°C.	1720°C.	1600°C.	1570°C.	—	—
Failure . .	—	1540°C.	over 1720°C. (28 lb. per sq. in.)	1630°C.	1620°C.	—	—
(50 lb. per sq. in.)	—	—	—	—	—	—	—
Thermal shock resistance.	30+	7	30+	30+	30+	30+	2
No. of reversals . .	—	—	—	—	—	—	—

well increase due to loss of temporary bonds, whilst the high initial crushing strength is lost on heating to some intermediate temperature, say, 900°C. The particular sample showed a low thermal shock resistance, but unfired bricks capable of withstanding 30 reversals can readily be obtained. Although in Great Britain and on the Continent use of such bricks in steelplants is still relatively limited, their popularity in the United States, where they have replaced at least half the fired products, leads one to believe that they will ultimately be used all over the world.

The properties of metal-cased magnesite tubes as used particularly for arc furnace side walls, are even more difficult to determine, in that the metal is a fundamental part of the structure. Rammed material cut from the centre of such tubes shows a porosity of the order of 20 per cent. and an after-contraction of less than 2 per cent. after 2 hours at 1500°C. It is clear that the properties of such tubes can vary just as those of unfired magnesite bricks according to the raw materials and methods of manufacture employed.

Other properties of magnesite brick of interest to steelmakers are as follows:

#### THERMAL CONDUCTIVITY

The conductivity of magnesite bricks is very high, as everyone knows who has walked close to even a new back wall built of straight magnesite bricks. At 300°C. mean the conductivity may be as high as 25 B.Th.U., but this value falls off considerably with rising temperature (*see* fig. 138, page 309).

#### ELECTRICAL CONDUCTIVITY

At room temperatures the electrical conductivity of magnesite bricks is quite low, but at higher temperatures, say 1500°C., it can be quite substantial. Use is made of this fact in certain furnaces, *e.g.*, the Greaves Etchells type arc furnace where the bottom constitutes the third electrode. In induction furnaces excessive conductivity would give rise to a "strike to the coil", but fortunately the part adjacent to the water-cooled coil remains relatively cold and usually unsintered. Trouble due to conduction is more likely to be associated with excessive moisture in the lining or, towards the end of the life, metal penetration.

#### THERMAL EXPANSION

The thermal expansion of magnesite bricks in the temperature range 20° to 1000°C. is rather high, *viz.*: about 1.2 to 1.4 per cent. Furthermore its expansion, unlike that of silica, continues until a temperature



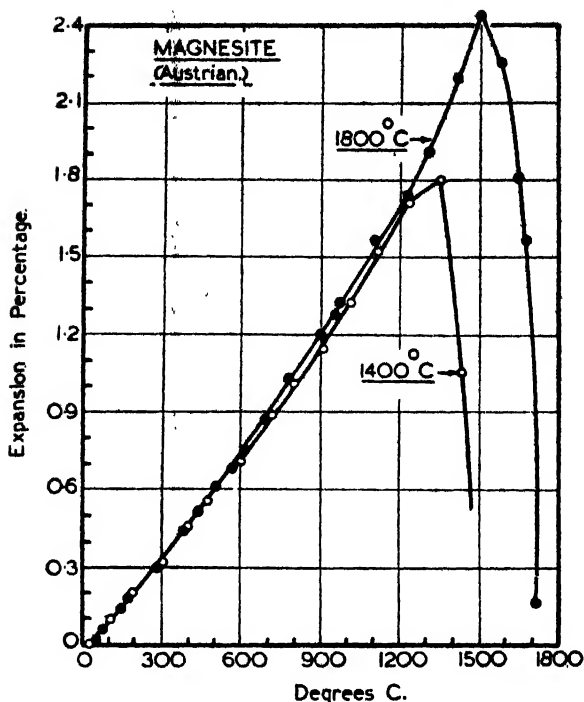
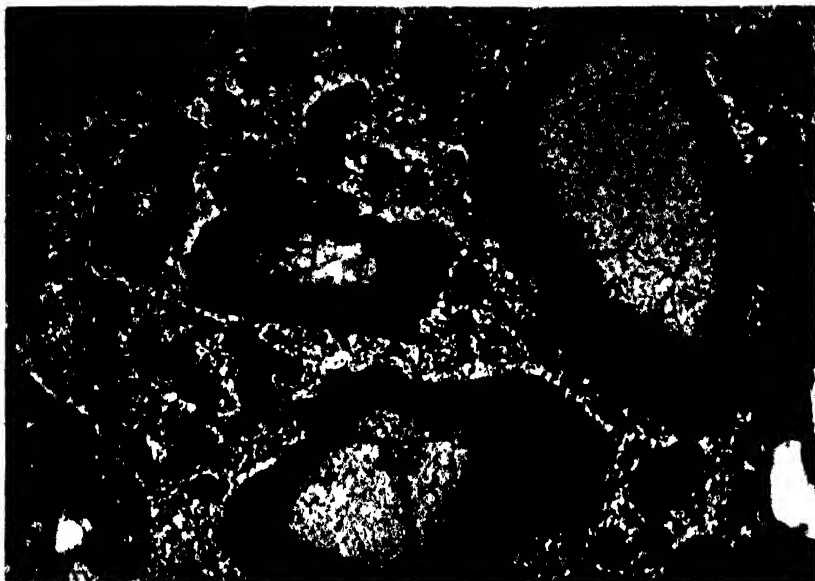


Fig. 75. Thermal expansion curves for Austrian magnesite, showing the increased stability produced by pre-firing to 1800°C. (after Heindl).

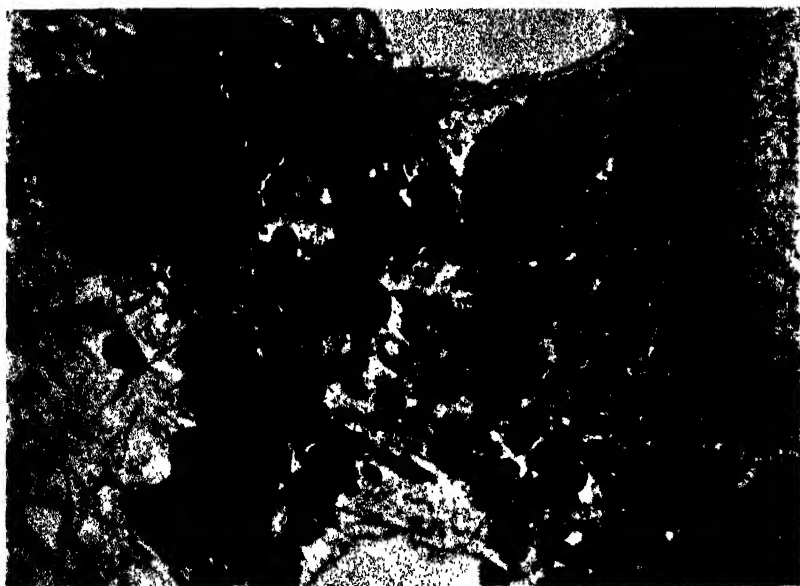
of the same order as that used in the initial firing is reached. Heindl's curves, as reproduced in fig. 75, show considerable shrinkage starting at about 1400°C. on a test-piece fired at this temperature, but no shrinkage until over 1500°C. on a second testpiece of Austrian magnesite refired at 1800°C. The sudden change from an expansion to a contraction is, of course, not due to the thermal expansion characteristics of the mineral, but to the onset of after contraction, the grains of magnesia being drawn together by the surface tension of the molten bond.

#### SLAG RESISTANCE

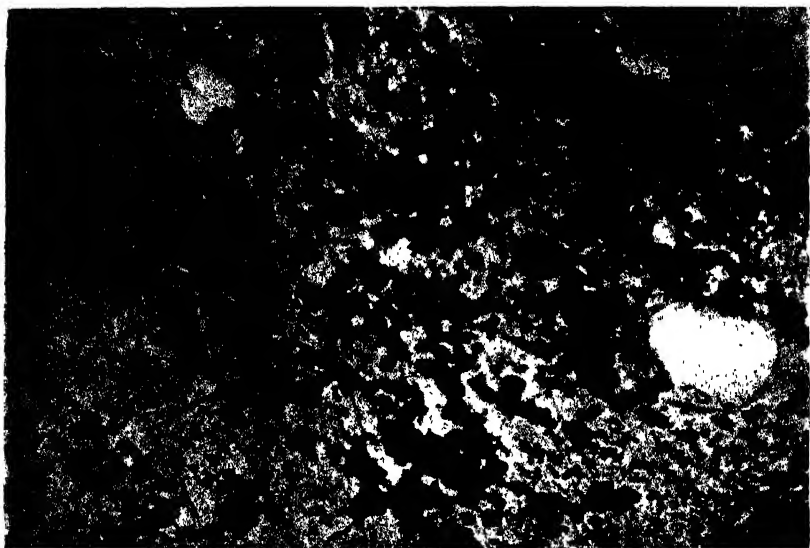
There is little doubt that magnesia will ultimately find a far wider field of application in steelplants, if only due to its remarkable resistance to chemical attack by slags rich in iron oxide and lime. Wear of magnesite bricks in exposed positions in open hearth furnaces is almost always due to the falling away of the brick ends, say in lumps of 2 in. thickness, rather than to slag attack as such. The photomicrographs



*Fig. 76. Grecian magnesite induction furnace lining, showing absorption of iron oxide by periclase crystals. Ordinary light  $\times 105$ .*



*Fig. 77. Grecian magnesite grain in induction furnace lining breaking up as a result of slag attack. Ordinary light  $\times 105$ .*



*Fig. 78. Grecian magnesite grain in induction furnace lining broken into small units as a result of slag attack. Ordinary light  $\times 105$*

shown in fig. 76 to fig. 78, pages 155 and 156, do, however, illustrate that under certain conditions the magnesite itself can be attacked but that even here the real weakness lies in the bond material. It will be seen that grains of Austrian magnesite tend to break down into a series of rounded crystals, which float out into the slag long before they have dissolved.

#### REFRACTORINESS-UNDER-LOAD

Whereas fireclay bricks tend to deform over a long range, behaving in a vitreous manner, magnesite bricks normally show little sign of failure to within a few degrees of the final fail point and then collapse suddenly. This is doubtless because they consist of a very refractory material—magnesia—surrounded with a bond of the forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ) or monticellite ( $\text{MgO} \cdot \text{CaO} \cdot \text{SiO}_2$ ), which are solid at one temperature and fully liquid only a few degrees higher. Booth, in his paper on the influence of chemical composition on the properties of magnesite and chrome-magnesite bricks, has attempted to calculate the mineral constitution of bricks of varying chemical analysis and related the initial fail temperature in a 28 lb. per sq. in. test to the calculated temperature of melt formation. The agreement obtained was remarkably good, the average difference between the 2 figures on 4 samples of clinker being only  $25^\circ\text{C}$ .

Fig. 79 shows characteristic curves for these materials, and also for silica which also generally fails rather suddenly, but at a temperature far closer to its melting point.

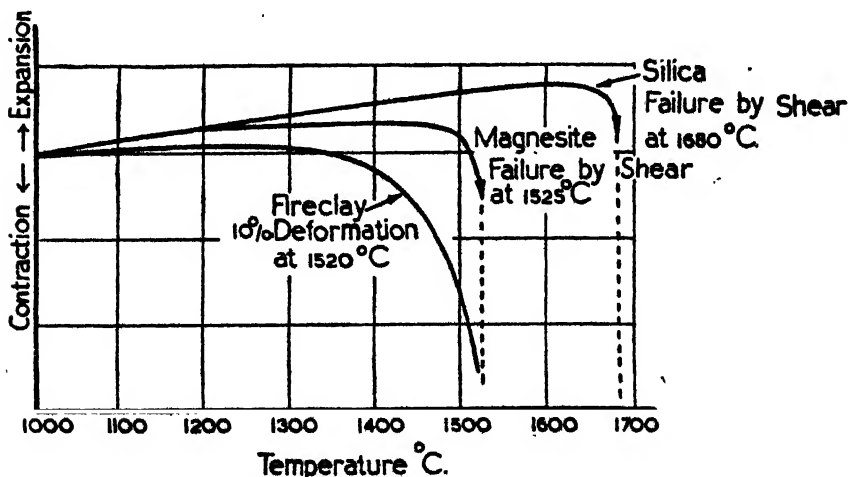


Fig. 79. Rising temperature refractoriness-under-load tests on magnesite, fireclay and silica bricks (50 lb. per sq. in.—10°C. per min. rate of rise).

## PERMANENT LINEAR CHANGE ON REHEATING

In general after-shrinkage tests on magnesite bricks are made for 2 hours at 1500°C., but there is little doubt that for use in certain exposed conditions, say, in open-hearth furnaces, much higher firing temperatures, say, 1700°C., should be employed. Many magnesite bricks are only fired at 1400°C., and above this temperature show appreciable shrinkage, but others are alleged to be fired at temperatures as high as 1650°C. Even these may, however, show shrinkage of the hot end in service due to the entry of iron oxide which promotes shrinkage and often causes the porosity of the working face to fall very considerably.

## SPECIFICATIONS

The only national specification for magnesite bricks known to the author is that employed for open-hearth furnaces in Russia (OST.5888). This states that the bricks must have strong edges and corners and give a clear ring when struck with a hammer. They should not contain more than 3 per cent. lime, or  $3\frac{1}{2}$  per cent. silica, and should have a specific gravity of over 3.50. The collapse point under a load of 2 kg. per sq. cm. should not be less than 1500°C., whilst the cold crushing strength should be over 300 kg. per sq. cm. and a bulk density not less than 2.6 g.p.ml. This specification would not be particularly difficult to meet for example with sea-water magnesia, but it should be stressed that any specification requires to be related to a particular and more specific job than merely the "open-hearth furnace", and that properties less capable of specification are always liable to prove more important than those actually included.

## APPLICATIONS

The main field of application for magnesite bricks in steelplants still lies in the hearths of basic arc and open-hearth furnaces. Substantial quantities are also used in the lining of barrel type inactive mixers, where bricks of particularly low porosity are usually specified. Magnesite bricks are also used to a limited extent in back walls, front walls and end walls of furnaces, and more recently in the top courses of the checker setting. For most of these purposes, however, bricks in which part at least of the magnesia is replaced by chrome ore are preferred. For arc furnace side walls, considerable use is, however, made of magnesite, generally in the form of round tubes, or metal-cased squares. The construction so obtained is particularly strong and is not as sensitive either to thermal shock or to damage by impact as the usual chrome-magnesite or magnesite brick structure.

The changes undergone by magnesite bricks in service are complicated, and depend very much on the particular position in which they are employed. The phenomena described by Wells and Van Vlack in connection with 96 per cent. MgO bricks used in the roofs of an all-basic furnace and in a turbo-hearth are, however, very characteristic of the position as a whole. These authors, like others before them, have observed the migration of liquid forming oxides away from the hot face, together with the absorption of iron oxide and other fluxes from the furnace atmosphere. They find that the iron oxide together with manganese oxide tend to dissolve in the periclase crystals at the working face, whereas the fluxes, notably silica, lime and phosphorous pentoxide, enter the silicate, forming a network around the magnesia. Although the magnesia crystals themselves tend to grow, the structure as a whole becomes weaker, due to the separation of the magnesia by low melting point material. The major loss generally occurs due to spalling several inches behind the hot surface, possibly due to the weakening of the brick at this point by a concentration of silicate liquid.

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## *Chapter IV*

# DOLOMITE

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SINCE DOLOMITE is the most readily available basic material of high melting point, it would seem to be the logical starting point for the development of steelplant refractories. In its calcined form it has done valiant service for over half a century in the open hearth, the bessemer and, more recently, the arc furnace. During World War II a considerable increase in relevant knowledge occurred as a result of the work done by the Basic Furnace Linings Committee, which was founded to study dolomite in its refractory applications. The problem was an urgent one, since chrome and magnesite were in short supply and furnace conditions particularly difficult as a result of the holding of casts during air raid alarms. The very full reports of the work of this Committee, as published in the Iron and Steel Institute Special Reports Nos. 33 and 35, will be freely drawn on in the present text.

The obvious corollary, that bricks made from this material should be equally useful and relatively cheap, has been realised, at least in part, in Great Britain, though strangely enough very few are produced in other parts of the world. The principal reason would appear to be the very tight control needed in manufacture, dolomite bricks casually made usually suffering from one or other of the following faults:

1. A tendency to "perish" on standing due to reaction of free lime with moisture in the air.
2. The tendency of  $\beta$  dicalcium silicate formed at high temperatures either in brickmaking or service to invert to  $\gamma$  dicalcium silicate on cooling with a 10 per cent. volume increase. This reaction which is well known to arc furnace users as falling slag causes even a strong brick to disintegrate into material of the fineness of face-powder and is frequently referred to as "dusting".

Because both "dusting" and "perishing" lead to the formation of fine dust, the two causes of failure have frequently been confused. Indeed it was not until the late Dr. Jay carried out the X-ray examination described later that the real cause of failure was appreciated and steps taken in the manufacturing process to deal with it.

Another ambiguity frequently associated with dolomite lies in the use of the term "stabilisation" to cover three quite different procedures, *viz.*:

1. The coating of calcined dolomite, *e.g.*, with pitch, to *reduce* the rate at which "perishing" occurs.
2. The conversion of the free lime to a silicate or ferrite to obviate hydration during subsequent storage and use.
3. The addition of boric acid, phosphates, or other "stabilisers" to prevent the inversion of  $\beta$  to  $\gamma$  dicalcium silicate.

It would appear better to use the word "stabilisation" solely for the last two procedures, which are carried out simultaneously in the manufacture of stable dolomite bricks. Bricks made by coating calcined dolomite grains either with a glassy bond or with organic materials, such as pitch, are better referred to as "semi-stable", since their life in storage is limited, particularly if the bricks are damaged in handling.

### THE MIXTURE $\text{CaO-MgO}$

When a true dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) is fully calcined and all the carbon dioxide driven off, the product is simply a mixture of lime and magnesia. This is shown by the Debye-Scherrer X-ray photographs, which consist of the superimposed lines of the two oxides, together with those due to traces of impurity. Such material is more reactive to moisture than magnesia, though much less so than lime itself. Its properties are those that might be expected from such a mixture. Those of magnesia have already been described (Chapter III); lime is similar but has a greater avidity. Thus when calcined dolomite is heated in contact with silica or ferric oxide, it is the lime which combines rather than the magnesia. Like magnesia, lime is a cubic mineral, having a specific gravity of 3.08 to 3.30. In its purest form its specific gravity is said to be 3.32, its refractive index 1.83 and its unit cell edge length 4.799 Å.

### BINARY SYSTEMS

#### $\text{CaO-MgO}$

The outline of this very simple system was given by Rankin and Merwin in 1916 (fig. 80). It will be seen that the lowest melting point composition still has a refractoriness of 2300°C., which puts it in the super refractory class.

#### $\text{CaO-SiO}_2$

This system has already been discussed in Chapter II in connection with the bonding of silica. From the standpoint of dolomite it is merely

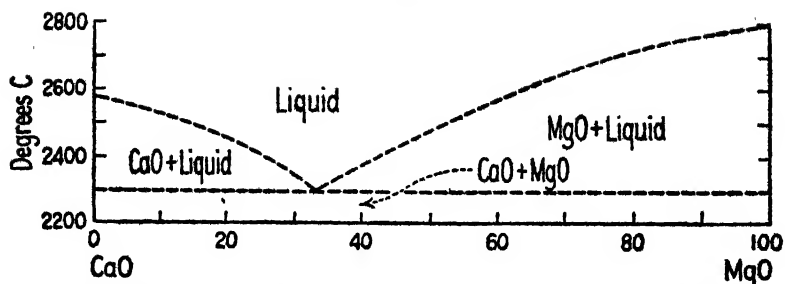


Fig. 80. System CaO-MgO (after G. A. Rankin and H. E. Merwin).

a binary section through the all important ternary system CaO-MgO-SiO<sub>2</sub>.

#### CaO-Fe<sub>2</sub>O<sub>3</sub>

When dolomite is used in a steel furnace it is always confronted with iron oxide. This may be present in its concentrated form as molten scale, iron oxide fume or as a slag constituent. Two of the reaction products frequently formed are di- and mono-calcium ferrite ( $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$  and  $\text{CaO} \cdot \text{Fe}_2\text{O}_3$ ), see fig. 81. The former frequently forms a solid solution with brownmillerite ( $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ ). Monocalcium ferrite shows a characteristic red colour in thin section similar to that of Grecian chromite and appears even brighter under crossed nicols. The dicalcium compound on the other hand is brown. As will be seen from the phase diagram (fig. 81) the melting point of the di- and mono-ferrites are roughly 1400° and 1200°C. respectively. It is not surprising, therefore, that iron oxide is capable of dissolving the lime in calcined dolomite at steelmaking temperatures.

#### TERNARY SYSTEMS

##### CaO-MgO-SiO<sub>2</sub>

This system, fig. 82, which was originally worked out by Ferguson and Merwin, and revised in 1943 by Osborn, is the basis for full-scale manufacture of stabilised dolomite bricks. The present-day batch consists essentially of a mixture of tricalcium silicate and magnesia. Compositions having a higher lime/silica ratio tend to show free lime, whilst those of lower lime/silica ratio usually contain dicalcium silicate, and must be stabilised to avoid  $\beta$  to  $\gamma$  inversion. The diagram also illustrates that any substantial pick-up of silica, such as might occur due to drip from a silica roof, will soon bring the melting point of the mixture down to open-hearth temperatures.

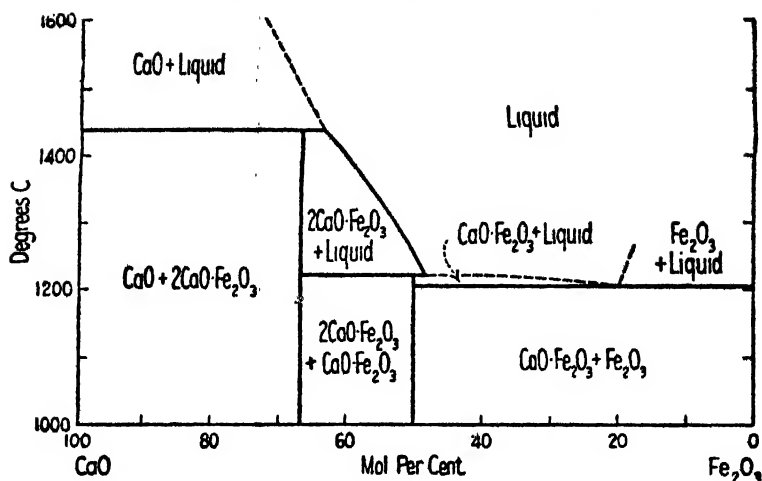


Fig. 81. System  $\text{CaO}-\text{Fe}_2\text{O}_3$  (after R. B. Sosman and H. E. Merwin).

### $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

This system (fig. 120) shows that the absorption of, say, a 40 : 60 alumina-silica mixture by tricalcium silicate results in a rapid drop in melting point, indicating the risks run by heating dolomite and fireclay brick in contact to temperatures of, say,  $1300^\circ\text{C}$ . or above. It also shows that if high refractoriness-under-load is to be achieved, dolomite batches should be as low in alumina as possible.

### $\text{CaO}-\text{MgO}-\text{Al}_2\text{O}_3$

This system illustrates the fluidifying action of alumina on dolomitic compositions.

### $\text{CaO}-\text{Fe}_2\text{O}_3-\text{SiO}_2$

A study of this system, figs. 41 and 42, shows that iron oxide has a marked fluidifying action on lime-silica melts. It is interesting to note that no ternary compounds of lime, silica and ferric oxide appear to have been discovered.

### $\text{CaO}-\text{Al}_2\text{O}_3-\text{Fe}_2\text{O}_3$

This system, which contains the rather interesting compound brownmillerite ( $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ ) has still not been worked out in detail. Brownmillerite itself is frequently found as a reaction product in slagged dolomite bricks and hearths.

## QUATERNARY SYSTEM



The cement chemists, *e.g.*, Lea and Parker, have carried out, and still are carrying out, a great deal of detailed work on this system, which is particularly important to them. It is also of interest to a dolomite brick manufacturer, though what the latter really requires is a complete evaluation of the quaternary system containing the above oxides together with magnesia. It is likely to be a long time before this information is available, and in the meantime research workers must be content to study the particular parts of this system with which they are concerned.

## RAW MATERIALS

## (a) AS QUARRIED

Dolomite, which got its name from Dolomieu, who first described it in 1790, was shown by Retgers in 1891 to have the chemical composition  $\text{CaCO}_3 \cdot \text{MgCO}_3$ . If formed in solution the deposit is primary, and similar to that of the Permian system in Great Britain, which runs in a broad band up the eastern side of the Pennines from Nottingham in the south to Coxhoe and Hartlepool in the north. If on the other hand it is formed by alteration on a deposited limestone, it will have irregular and discontinuous boundaries, as found in the quarries of north and south Wales. Most of the British dolomites used in the manufacture of fettling material contain approximately equimolecular proportions of calcite and magnesite in solid solution. X-ray examination shows those high in lime to be mixtures of dolomite and calcite.

The pure mineral is white in colour, and has a specific gravity of 2.85. Impurities such as carbonaceous matter and iron compounds may result in the material varying from pale buff to red, blue or even black. Typical analyses and the physical properties of several dolomites are given in Table XII, from which it will be seen that the primary dolomites tend to be purer, more porous, and therefore softer than the dolomites of secondary origin.

In the United States the great distance between dolomite quarries and the steelworks has resulted in the use of a considerable amount of magnesite, but dolomite, both raw and prepared, does, nevertheless, play a very important part in the fettling of open-hearth furnaces.

## (b) PRODUCTION OF DOLOMA

The term "doloma" was finally recommended by the Basic Furnace Linings Committee after considering numerous alternatives. It is



TABLE XII  
COMPOSITION AND PROPERTIES OF SELECTED DOLOMITES

Origin and formation	Chemical analysis. %						Physical properties			Remarks
	Silica SiO <sub>2</sub>	Mixed oxides R <sub>2</sub> O <sub>3</sub>	Lime CaO	Magne- sia MgO	Loss on igni- tion	Total	Specific gravity	Bulk density g. per ml.	Porosity %	
<i>S. Yorks, Derby and Notts:</i> Permian lower magnesium limestone	0.33 0.74 0.87	0.52 0.72 0.60	30.63 30.25 30.32	21.50 21.28 21.23	47.37 47.00 47.13	100.35 99.99 100.15	2.84 2.84 2.85	2.47 2.41 2.39	13.0 15.1 16.3	Soft
<i>Durham:</i> Permian system	0.89 0.75	0.96 0.70	30.6 31.1	20.6 20.4	46.95 46.80	100.00 99.75	2.85 2.85	2.53 2.51	11.2 12.3	Medium to soft
<i>Leicestershire:</i> Car- boniferous lime- stone	0.46 2.06	1.80 1.64	31.08 30.80	20.42 20.21	46.65 46.06	100.41 100.77	2.81 2.83	2.54 2.68	9.6 5.3	Medium to hard
<i>North Wales:</i> Car- boniferous lime- stone	0.92 2.06	1.48 0.83	31.10 31.78	20.20 19.35	46.29 45.94	99.99 99.96	2.85 2.87	2.74 2.68	3.8 6.2	Hard
<i>South Wales:</i> Car- boniferous lime- stone	0.21 1.28 1.87	3.62 0.81 1.14	30.82 32.48 31.08	19.64 19.41 20.26	46.36 45.15 46.06	100.65 99.13 100.41	2.87 2.82 2.84	2.75 2.77 2.79	4.3 1.8 1.7	Very hard
<i>Scotland:</i> Appin limestone forma- tion	3.70 3.30	1.66 0.52	28.9 29.1	20.85 21.75	44.80 45.47	99.91 100.14	2.87 2.84	2.85 2.77	0.5 1.2	Friable
<i>Dolomite (theoreti- cal)</i>			30.41	21.87	47.72	100				

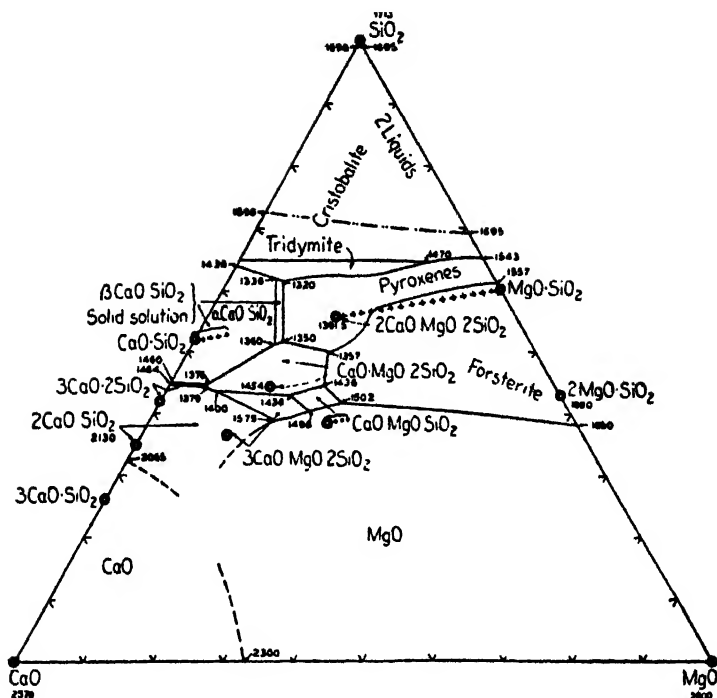


Fig. 82. System  $\text{CaO-MgO-SiO}_2$  (after E. F. Osborn).

intended to express the fact that this material consists of the oxides formed from dolomite in the same way that magnesia indicates the oxide product from magnesite. The term is now largely replacing the original British word "basic", whose use frequently led to confusion. Thus a basic hearth might be a hearth made from basic, *i.e.*, calcined dolomite, or a magnesia hearth which behaves in a chemically basic manner.

The production of doloma consists essentially in carrying out the following reaction:—



If, however, this reaction is stopped immediately the  $\text{CO}_2$  has been driven off, the product is too reactive and porous for use in the steel-plant. The calcination must, therefore, be carried to a much higher temperature, of the order of  $1700^\circ\text{C}$ ., when the porosity drops to about 15 per cent. and the product is capable of being stored for several weeks without serious deterioration. The effect of firing temperature on the

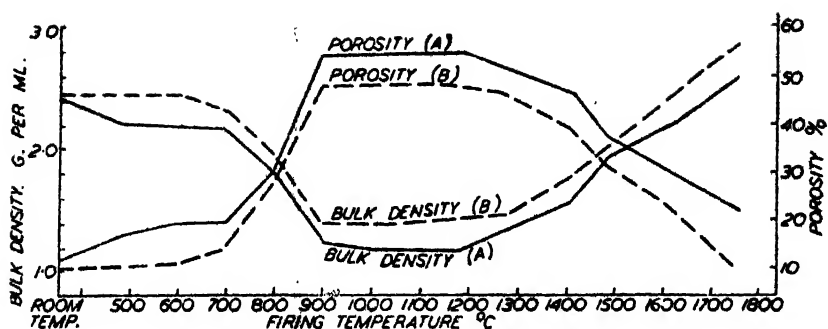


Fig. 83. Bulk density and porosity of doloma prepared in the laboratory (after Bradley, Chesters and Douglas).

bulk density and porosity of doloma is shown in fig. 83, from which it will be seen that under the conditions used in the laboratory, which were intended to simulate the rates of heating obtained in commercial cupolas, a temperature of over  $1700^{\circ}\text{C}.$  was necessary to achieve the bulk density of 2.5 g.p.ml. normally considered necessary for fettling material. It will be seen that both the porosity and bulk density are influenced by the nature of the raw material, the two dolomite samples, A and B, used having the following compositions:

	A	B
Silica $\text{SiO}_2$ .. .. .	0.46%	3.68%
Mixed Oxides $\text{R}_2\text{O}_3$ .. .. .	0.52%	0.81%
Calcium Carbonate $\text{CaCO}_3$ ..	54.25%	51.21%
Magnesium Carbonate $\text{MgCO}_3$	44.52%	43.39%
	<hr/>	<hr/>
	99.75%	99.09%

Incidentally it will be seen from fig. 84, which shows diagrammatically the results of X-ray examination that the dissociation of dolomite results in the intermediate formation of calcined carbonate, but that heating to  $900^{\circ}\text{C}.$  leaves nothing but magnesia and lime as the product. On further heating these oxides undergo crystal growth, the eventual size being of the same order in both cases but still extremely small. The apparent solid density of doloma unlike that of magnesia, is of little assistance in assessing firing temperature, since it remains roughly constant at approximately 3.4 once a temperature of  $1000^{\circ}\text{C}.$  has been reached.

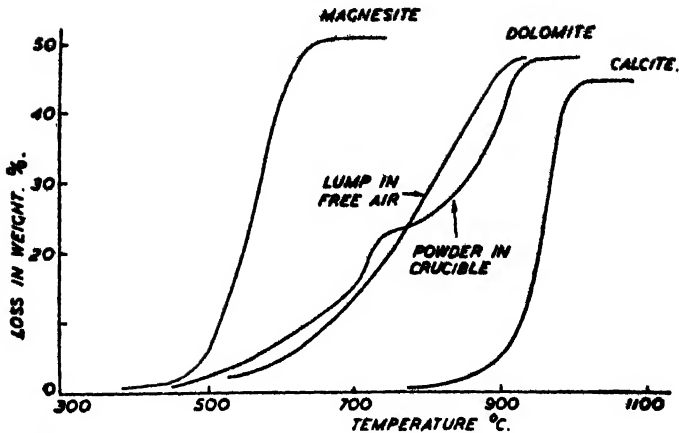


Fig. 84. Loss in weight/temperature curves for magnesite, dolomite and calcite (after Bradley and Jay).

Most of the doloma used in Great Britain is produced either in cupolas (*see* fig. 85, p. 181) or vertical shaft kilns. Such material is considered to possess at least one advantage over the rotary kiln product, *viz.*, a higher angle of rest when crushed. In the cupola or kiln practice the fuel and raw stone are charged in alternate layers and the product is, therefore, contaminated with ash and incidentally, therefore, with sulphur. The presence of these impurities may well play a part in the production of a fully shrunk product. The operation of such plant has much in common with that of the blast furnace, in that it is important to maintain an even distribution of gas flow, which can only be achieved by careful sizing and charging of the raw materials. The processes which occur may be considered as divided into four stages, *viz.*, drying, calcination—yielding a porous mixture of lime and magnesia, burning—in which the porosity is greatly reduced and temperatures of the order of 1800°C. or over achieved, and finally cooling—which mainly serves to preheat the incoming air blast and thus increase the maximum temperature attained in the burning zone. After the material has been fired, it is usually picked over on a conveyor belt, any soft-fired material being rejected.

In the United States, dolomite is normally calcined in rotary kilns with an addition of iron oxide which both speeds up the sintering and increases the stability of the product. An  $\text{Fe}_2\text{O}_3$  content of 5 per cent. or more in the final material is not at all unusual. Details of the procedure used have been described by H. C. Lee, who states that where the calcining plant is close to the user, quite low temperatures (about

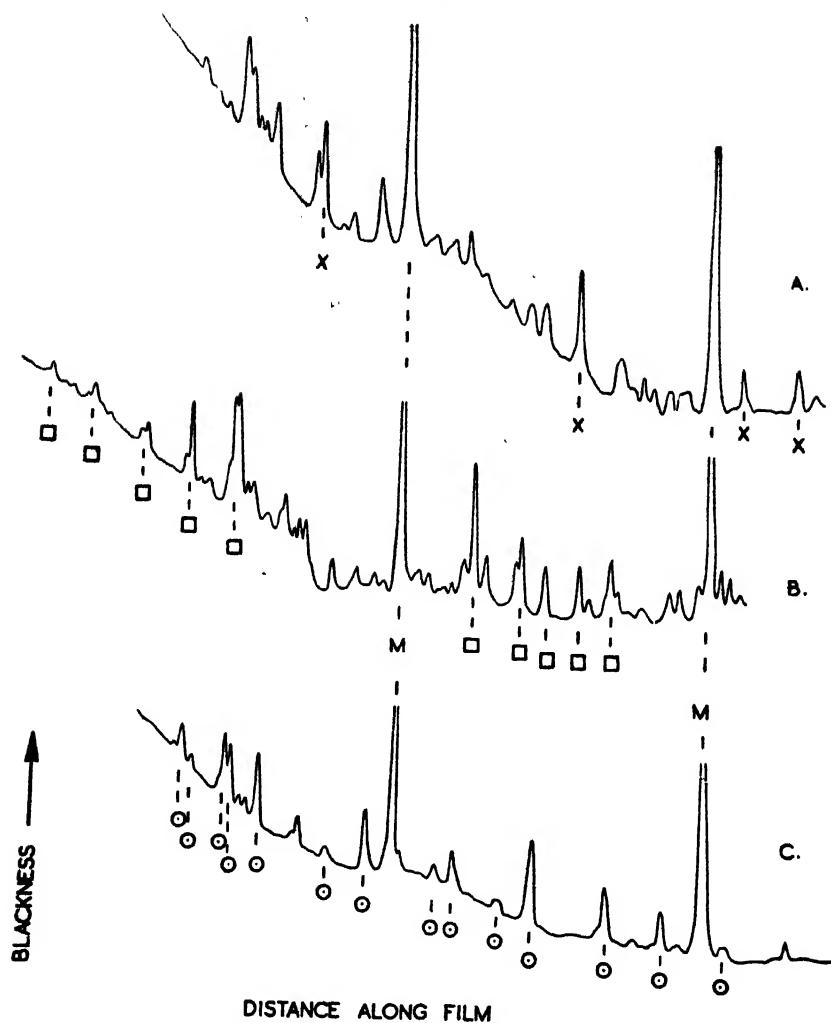


Fig. 86. Photometer curves of X-ray (Debye-Scherrer) photographs.

A. Unstable dolomite brick containing  $\text{CaO}$  (X).

B. Dusted dolomite brick containing  $2\text{CaO}-\text{SiO}_2$  (□).

C. Stable dolomite brick consisting of  $3\text{CaO}-\text{SiO}_2$  (○) and magnesia (M).

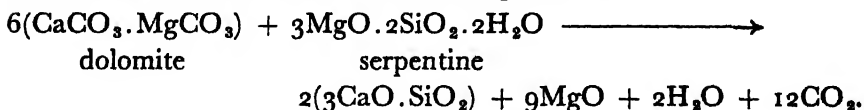
1100°C.) are often employed. The more important product is, however, that made in the rotary kiln, and particularly in Ohio, whose output is more than all that of the other States combined. A typical product containing 7.5 per cent. of iron oxide is said to have a porosity of 7½ per cent. and a grading range of ½ in. to 120 mesh.

## (c) PRODUCTION OF STABILISED DOLOMITE CLINKER

The ceramic literature of the last 30 years contains numerous suggestions as to how dolomite can be stabilised or the lime fraction removed. Certain of the methods suggested are almost fantastic, whilst others although showing promise still fall short of being a commercial proposition. Among the first successful experiments reaching the works stage were those of Tyler and Rees, who in 1934 produced a brick consisting essentially of 75 per cent. dolomite and 25 per cent. sudanite—a mineral containing a mixture of magnesite and talc.

Although in the present brick use is made of serpentine instead of sudanite, and great improvements have taken place in the method of clinker production, grading, pressing, drying and firing, the brick used today is fundamentally similar to that produced in 1934. These early bricks were made by firing clots of suitable composition followed by regrinding, moulding in a small hydraulic press, and firing in a pilot kiln. They showed a serious brick to brick variation and suffered from hydration in storage and "dusting" in service.

Numerous suggestions had been made as to the cause of these difficulties, but it was Dr. A. H. Jay of the Central Research Department (The United Steel Companies Limited) in collaboration with Sir Lawrence Bragg (then at Manchester University) who showed that the hydrated bricks originally contained uncombined lime due to insufficient silica content, or inadequate mixing and firing, whilst those which "dusted" contained considerable amounts of  $\gamma$  dicalcium silicate (see fig. 86). It was as a result decided that the batch composition should be adjusted so as to achieve the following reactions:



Since the control would have to be extremely precise to ensure that tricalcium silicate was the sole calcium compound produced, it is considered safer to operate slightly on the silica-rich side, any dicalcium-silicate which may form being stabilised by the addition of small amounts of stabiliser, such as boric acid or phosphates.

The very extensive literature on stabilisation has been reviewed by Seil and his staff, whilst many other workers have carried out systematic studies, by adding increasing amounts of such materials as serpentine, olivine, iron oxide and zirconia, to raw dolomite, thus fig. 87 and Table XIII show the results obtained by Rait and Green, from which it will be seen that they found it necessary to add 15 per cent. of serpentine to reduce the free lime content to zero.

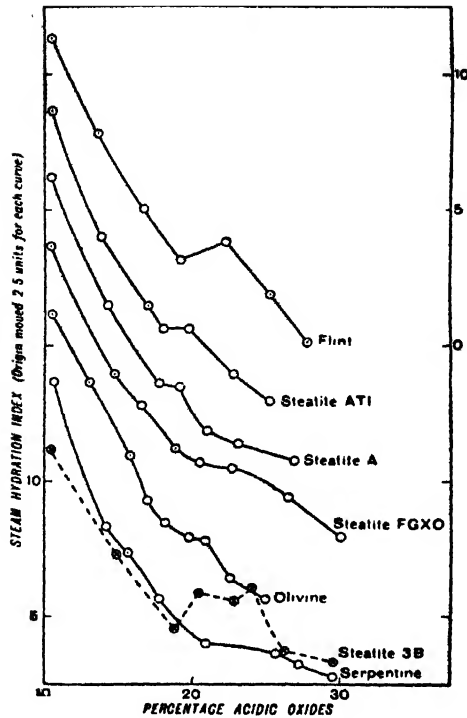
TABLE XIII

COMPARISON OF CALCULATED AND DETERMINED  
FREE LIME CONTENTS*(Rait and Green)*

<i>Composition of mix</i>		<i>Percentage free lime</i>	
<i>Percentage Dolomite B</i>	<i>Percentage stabiliser</i>	<i>Calculated</i>	<i>Determined</i>
100	—	29.64	28.40
96	4 flint	8.36	8.26
95	5 steatite A.T.I.	18.12	16.28
96	4 steatite A	15.64	16.46
92	8 steatite A	4.04	4.39
95	5 steatite F.G.X.O.	17.41	18.16
90	10 steatite F.G.X.O.	5.75	5.88
95	5 steatite 3B	14.51	16.40
92	8 olivine	8.89	8.18
90	10 olivine	5.03	5.09
95	5 serpentine	18.60	19.16
93	7 serpentine	14.37	14.65
90	10 serpentine	8.15	7.00
85	15 serpentine	0.0	0.0

More recently two teams, Newman and Wells, and Pole, Beinlich and Gilbert, have worked on the allied problems, the former dealing particularly with the influence of various additions on the  $\beta$ - $\gamma$  inversions of dicalcium silicate and the latter on the effect of small additions (10 per cent.) of alumina, olivine, chromic oxide and zirconium oxide, on the stability of dolomite bricks. Their conclusions, though interesting, would not appear to affect at all materially the present procedure.

In actual practice the raw materials are first wet ground in a tube mill and then passed to a slurry tank, where they are kept stirred until fed into the top of a rotary kiln (fig. 88, p. 181). The kiln is normally fired with pulverised coal and attains a temperature of over 1600°C. As the slurry passes through it, it first loses water and then carbon dioxide from the dolomite. From then onwards reactions resulting in the formation of tricalcium silicate, magnesia and a small amount of glass, take place. Careful control is needed in the firing zone, since otherwise the pea-size clinker particles may unite to yield balls several inches in diameter. The redhot material leaving the lower end of the kiln passes



*Fig. 87. Relation between steam hydration index and percentages of acidic oxides for clinkers prepared from dolomite and various silicates (after J. R. Rait and A. T. Green).*

through a cooler and thence via elevators to crushers, after which it is sieved into fractions and stored in steel hoppers.

Numerous other processes have been tried out in different parts of the world, but the above procedure is the only method of producing truly stable clinker used on any large scale.

#### (d) PROPERTIES OF DOLOMA

Those who wish to study this field in detail are advised to read the Iron and Steel Institute Special Report No. 33, some of the contents of which are summarised below.



### 1. Chemical analysis

This is, of course, dependent both on the raw material and the ash pick-up from the kiln. The following, however, gives a rough impression of the general range of compositions for the Derbyshire, Nottinghamshire and Yorkshire product:

MgO	..	..	..	35—38%
CaO	..	..	..	53—59%
Fe <sub>2</sub> O <sub>3</sub>	..	..	..	1.7—2.5%
Al <sub>2</sub> O <sub>3</sub>	..	..	..	1.6—2.4%
SiO <sub>2</sub>	..	..	..	2.7—3.5%
S	..	..	..	0.07—0.11%

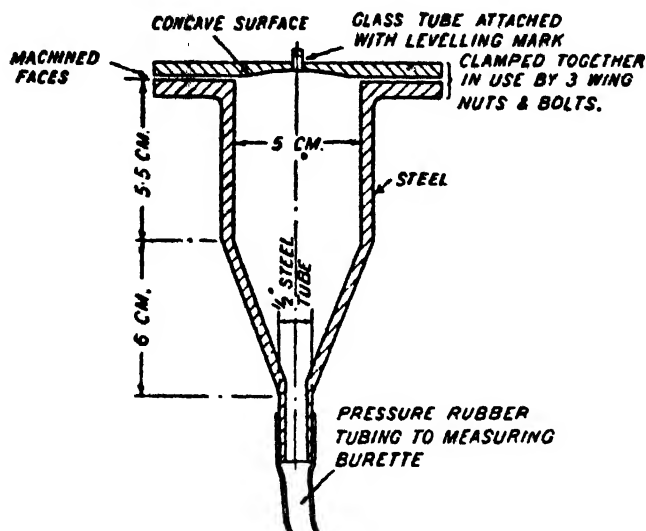
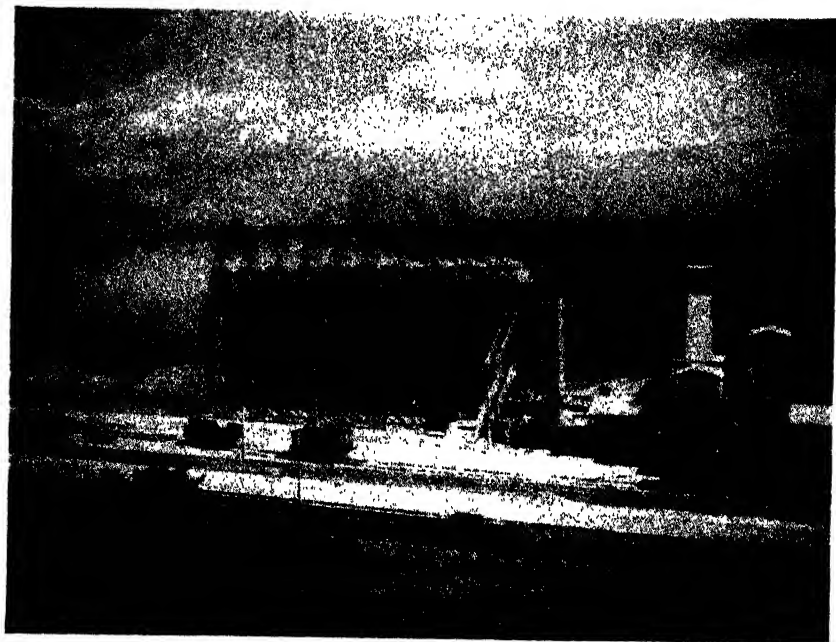


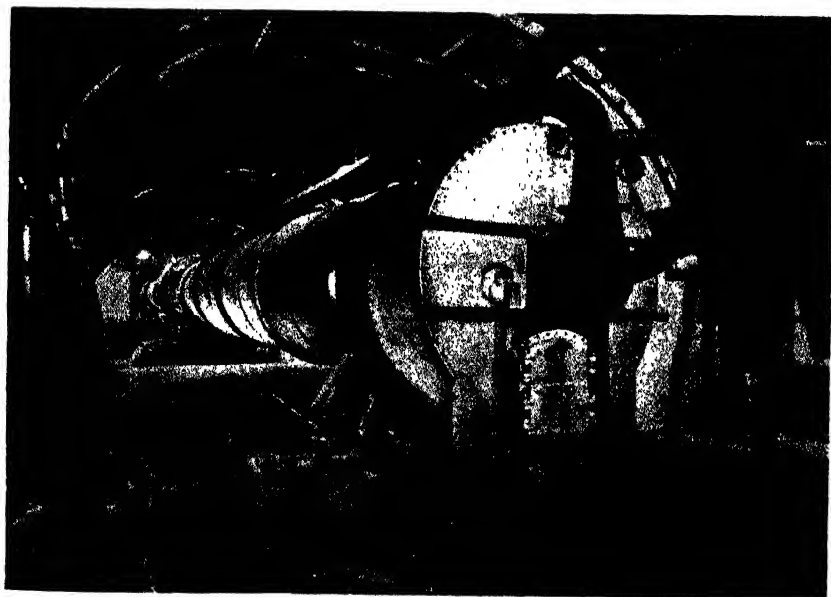
Fig. 89. Steel container used in determining bulk density of doloma by mercury displacement.

### 2. Porosity, bulk density and true specific gravity

A special apparatus, the principal part of which is shown in fig. 89, was developed for the rapid determination of the bulk density of doloma. It consists, as will be seen from the sketch, of a conical shaped vessel of 5 cm. diameter with a machined fitting lid. The sample to be tested, which is normally of a size similar to that employed in practice, is placed in the container, the lid attached, and the volume of a known weight determined by mercury displacement. Where desired the true porosity can be obtained by combining the bulk density figure with the specific gravity as determined for example by means of a Rees-Hugill flask.



*Fig. 85. Cupolas used for firing dolomite (Steelley Co. Ltd.).*



*Fig. 88. Rotary kiln used for production of stabilised dolomite clinker (Steelley Co. Ltd.).*



*Fig. 90. Hydraulic pressing of stabilised dolomite bricks (Steelley Co. Ltd.).*

### 3. *Hydration resistance*

Two methods have been examined: in the first the finely ground product is subjected to the action of steam for five hours and the percentage of hydration is determined by loss on ignition at 1000°C.—after drying. The best results appear to be obtained when a relatively coarse fraction, for example,  $\frac{1}{2}$  in. to  $\frac{1}{4}$  in., is employed. It was shown as a result of a large number of tests that there was a good correlation between the results obtained in this test and the actual crumbling observed on standing as estimated by the percentage passing a 240 mesh screen. In the second test, samples of known grading are merely exposed in the atmosphere for long periods. Such tests reveal the importance both of high bulk density and large size in giving stability,  $\frac{1}{2}$  in. sample of average bulk density showing increase in weight of only 0.13 per cent. after 21 days, compared with 4.5 per cent. of the corresponding 25–72 mesh fraction.

### 4. *Grading and packing of aggregates*

Where the product is very coarse, packing density is best assessed by the use of a machine of the type developed by Westman and Hugill, and subsequently used by Chesters and Lea. Where, however, the material is  $\frac{1}{2}$  in. or less, use can be made of the standard AFA type rammer. Such a machine is particularly useful for controlled work on tarred material as used for open-hearth furnace and bessemer converter bottoms.

### 5. *Sintering tendency*

As would be expected from work with other materials, the sintering tendency of doloma is greatly enhanced by the fineness of grain and the presence of impurities.

### 6. *Angle of rest*

A great deal of work was carried out by the Basic Furnace Linings Committee on the angle of rest of doloma both dry and bonded, and under laboratory and works conditions. Work on a series of close ranges suggested that whereas with  $\frac{3}{4}$  in. material an angle of 42° was common, with finer material this decreased, for example, to 36° for 30–60 mesh. Given suitable combinations of coarse and fine, much steeper angles can be obtained, but in practice the user is faced with the problem of grain size segregation during throwing.

### 7. *Slag resistance*

The fusion relationships for mixtures of calcined dolomite-magnesite and basic slag have been studied by Dobbins and Rees, using a method in which mixes varying by 10 per cent. of each constituent were studied, the fusion points being determined by the method of glazing.

Surprisingly enough these authors found that there was little to choose between the slag resistance of doloma and magnesia, in spite of the fact that the melting point of lime compounds is generally lower than that of their magnesia equivalent. This conclusion would hardly seem consistent with the observation of many open-hearth workers, that the amount of doloma required to fettle a basic furnace bottom is normally two to three times the equivalent amount of magnesia.

#### (e) PROPERTIES OF STABILISED CLINKER

X-ray examination offers by far the best method of studying the constitution of clinker, but where it is desired to test for the presence of small amounts of free lime (less than 2 per cent.), other tests, notably White's test and the glycerol test, are preferred. In White's test a small amount of finely ground sample is placed on a microscope slide and wetted with a solution consisting of 5 g. of phenol dissolved in 5 c.c. of nitro-benzene with the addition of 2 drops of water. The slide is then examined between crossed nicols, using a magnification of about 150. The presence of free lime is shown by the formation of long highly bi-refrangent needles which radiate from the free lime particles. Where the lime content is high these crystals appear almost immediately, but where it is low it may be necessary to leave the slide for an hour or so after preparation.

Details of the method used in carrying out the glycerol test have been fully described by B. Bakewell and G. E. Bessey in their D.S.I.R. Building Research Report. As Table XIII shows, there is a close correlation between the amount of free lime expected from equilibrium considerations and that actually determined by the glycerol test.

Stabilised clinker, like doloma, can vary appreciably in bulk density. It is generally considered that an average figure of at least 2.4 g.p.ml. should be obtained for clinker of the type here described.

### MANUFACTURE OF DOLOMITE BRICKS

#### (a) SEMI-STABLE BRICKS

Some of the first dolomite bricks to give useful service in the open-hearth furnace were of the semi-stable type. They were made by grinding calcined dolomite with tar and ramming the mass in large moulds—a technique already well developed in connection with the production of basic bessemer bottoms. Such bricks were used almost immediately and no serious trouble was, therefore, experienced due to hydration. Somewhat more stable bricks can be made by adding a flux to the batch and firing in the ordinary way. The life of such bricks in storage

can be extended to, say, six months, by boiling them in tar after the firing process. The firing itself is quite difficult, there being a tendency for abnormally large volume changes to occur at quite low temperatures. The percentage of "wasters" is, therefore, liable to be high, though certain firms have now reduced the losses to a point which makes commercial production of such bricks, for example, for use in basic bessemer side walls, quite practicable.

(b) STABLE DOLOMITE BRICKS

For a long time the number of patented methods of stabilising dolomite bricks probably exceeded the number of stable bricks sold. From 1934 onwards the situation changed rapidly, when the Steetley Company demonstrated that dolomite could be stabilised commercially with serpentine, provided the technical control was adequate. Since then there has been a steady increase in the production of stabilised dolomite bricks in Great Britain, as many as five firms being employed in this activity during the Second World War. For various reasons the production of such bricks in other countries is still very small, though interest is still being shown in a number of quarters. The general procedure employed in Great Britain is briefly summarised below.

Clinker from the rotary kiln is ground and sieved out into two or three grain size fractions. These are then combined and mixed in a paddle type mixer, giving for example a grading of the 60-10-30 coarse-medium-fine type. The water content required for hydraulic pressing at 10,000-15,000 lb. per sq. in. (*see* fig. 90, p. 182) is quite low, usually about 4 per cent. Even so the drying of dolomite bricks can lead to cracking, both in the drier itself or in subsequent firing. The cause is somewhat obscure, but it is known that the trouble can be minimised if drying is done with a stream of dry clean air at a temperature below 60°C.

Firing is carried out in down draught kilns, either of the circular or rectangular type, using coal, oil, or producer gas as the fuel (*see* figs. 91 and 92, page 191). The temperatures used are similar to those normally employed with magnesite, *viz.*: 1350° to 1450°C., while a soaking period of at least 24 hours is considered desirable. If the soaking time is too short or the firing temperature too low, the bricks will contain a considerable amount of free lime, and be, therefore, liable to disintegrate in storage. Incidentally Carlson has shown that tricalcium silicate tends to break down when kept at 1200°C., yielding dicalcium silicate and free lime. At a rather higher temperature the products recombine, bricks fired at 1400°C. or over not showing any reaction in White's test or any tendency to crumble in storage. A convenient way

of checking firing treatment on the plant is afforded by repeated measurements of firing shrinkage, which should be, say,  $\frac{3}{16}$  in. on the 9 in. length of a standard square. It must be stressed that the above particulars apply to the processes at present employed in Great Britain, and not to the numerous alternatives developed in other countries.

### (c) COMPOSITE DOLOMITE BRICKS

Numerous attempts have been made to produce stable dolomite bricks with special properties, by blending such materials as magnesite and chrome with the stabilised clinker. Thus Douglas, Bradley and Chesters, showed that replacement of the intermediate grain size fraction in a stabilised dolomite brick by dead-burned magnesite resulted in a substantial increase in initial thermal shock resistance. Bricks in which the intermediate section is replaced by chrome ore have also given reasonable laboratory test data, but are not suitable for use, say, in backwalls, where the temperatures are high and softening of the relatively low melting point constituents occurs. During the Second World War the need to save magnesite led to the development of numerous intermediate products, *e.g.*, magnesite bricks in which 20–40 per cent. of the magnesia was replaced by stabilised dolomite clinker. Their use today is, however, very limited.

## PROPERTIES OF DOLOMITE BRICKS

### (a) SEMI-STABLE DOLOMITE BRICKS

The following analysis of a typical British brick of this class:

SiO <sub>2</sub>	..	..	..	..	4.3%
Fe <sub>2</sub> O <sub>3</sub>	..	..	..	..	2.5%
Al <sub>2</sub> O <sub>3</sub>	..	..	..	..	2.2%
CaO	..	..	..	..	51.7%
MgO	..	..	..	..	38.2%
Loss on ignition	..	..	..	..	1.1%

shows that it is essentially similar chemically to fettling doloma. Table XIV, column 1, gives the properties of a semi-stable dolomite brick, from which it will be seen that these compare favourably with those of other basic bricks. Thus the porosity is low, the refractoriness under load is at least moderate, whilst the thermal shock resistance is substantially better than that of stabilised dolomite brick, as is also its resistance to an iron oxide—lime slag at 1650°C. Such bricks still possess one serious limitation, *viz.*: that they start to disintegrate after about six months in storage, particularly if damaged in handling.

## (b) STABILISED DOLOMITE BRICKS

A selected analysis of a British brick in this class is as follows:

SiO <sub>2</sub>	..	..	..	14.4%
Fe <sub>2</sub> O <sub>3</sub>	..	..	..	3.4%
Al <sub>2</sub> O <sub>3</sub>	..	..	..	1.5%
CaO	..	..	..	40.0%
MgO	..	..	..	40.4%
Loss on ignition	..	..	..	0.3%

TABLE XIV  
PROPERTIES OF DOLOMITE BRICKS

	1	2	3
	<i>Semi-stable dolomite brick</i>	<i>Dolomite brick (X.6)</i>	<i>Dolomite brick (X.7)</i>
Porosity, per cent. ..	22.8	22.1	24.7
Bulk density, g.p.ml. ..	2.52	2.58	2.53
Apparent solid density g.p.ml.	3.27	3.31	3.36
Cold crushing strength—on end—lb. per sq. in. ..	3680	>8300	5090
Permeability to air—perp. 9 in. × 3 in. face—1 skin, c.g.s. units .. ..	0.29	0.088	0.12
Permanent linear change on reheating, 2 hr. 1500°C.	1.2%	0.0%	0.2%
Refractoriness-under-load— maintained temp. test— 25 lb. per sq. in. @ 1600°C.	—	Failed in 32 mins.	Failed in 11 mins.
Rising temp. test—initial softening .. ..	1360°C.	1540°C.	1460°C.
Rapid softening .. ..	1520°C.	1610°C.	1510°C.
Failure .. ..	1610°C.	1680°C.	1600°C.
(50 lb. per sq. in.)			
Thermal shock resistance.			
No. of reversals .. ..	23	2	3
Slag resistance (when tested with “4Fe <sub>2</sub> O <sub>3</sub> —CaO” slag at 1650°C.) .. ..	Definitely higher than stabilised dolomite brick. No bursting expansion with iron oxide	—	—



X-ray examination of such a brick (fig. 86) shows it to consist essentially of tricalcium silicate and magnesia.

Characteristic properties of two samples are given in Table XIV, from which it will be seen that the porosities are again quite low and the crushing strengths unusually high. There is good volume stability (two hours at  $1500^{\circ}\text{C}.$ ), whilst the refractoriness-under-load data are comparable with those of magnesite bricks. The thermal shock resistance, however, is unusually low at 2–3 reversals. Other properties of interest to the steelplant operator are as follows:

#### THERMAL CONDUCTIVITY

This runs at about 15 B.Th.U. at  $300^{\circ}\text{C}.$  (mean) compared with, say, 25 B.Th.U. for magnesite. The replacement of magnesite bricks in open-hearth or arc furnace bottoms by dolomite bricks is therefore equivalent in some small measure to insulation.

#### SPECIFIC HEAT

This is similar to that of most fireclay and silica bricks, being of the order of 0.25 over the temperature range  $20$ – $750^{\circ}\text{C}.$

#### THERMAL EXPANSION

This is normally about 1.3 per cent. for the temperature range  $20$ – $1000^{\circ}\text{C}.$ , *i.e.*, very similar to that shown by magnesite bricks.

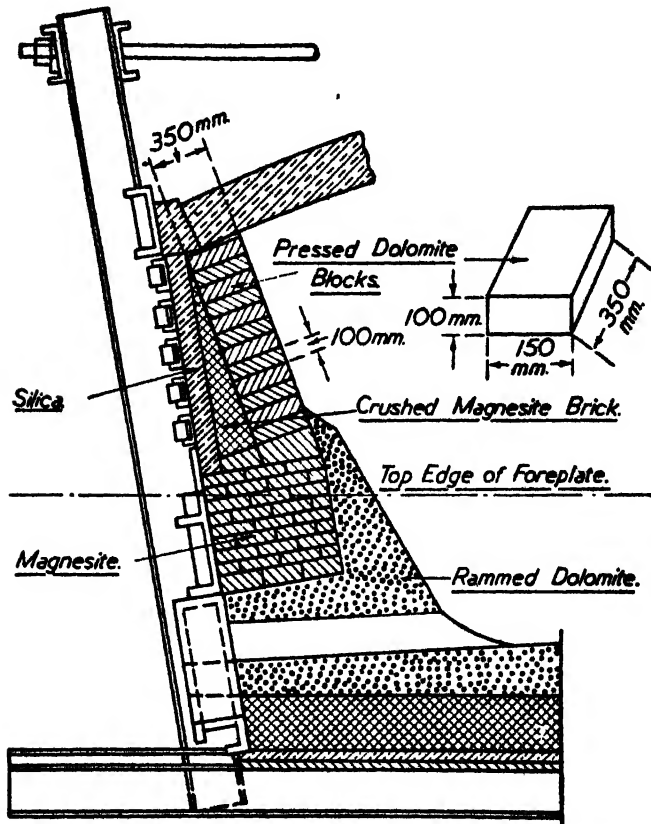
#### SLAG RESISTANCE

Although dolomite bricks have given service in open-hearth and arc furnace bottoms similar to that obtained with normal magnesite bricks, laboratory tests suggest that their resistance to attack by iron oxide rich slags is definitely lower than that of pure magnesia. This is brought out in fig. 93, p. 192, which shows the effect of heating up test blocks varying in composition from magnesite to stabilised dolomite with pellets of a 4–1 ferric oxide-lime mixture on the top surface.

#### APPLICATIONS

The first successful trials of stabilised dolomite bricks in Great Britain were carried out by The United Steel Companies Limited in 1934. They showed that this type of brick could replace magnesite, for example, in basic open-hearth furnace tap holes. Since then, and particularly since 1939, their use has extended to the whole of the

sub-hearth of fixed open-hearth furnaces and to the top layers of sub-hearths in tilting furnaces, their use in these positions having led to a substantial saving due to the lower price of the stabilised dolomite bricks. They are also extensively employed in the front, back, and end banks of fixed furnaces, in the splays of tilting furnaces, and in reheating



*Fig. 94. Back wall of 150-ton open-hearth furnace showing use of pressed (unfired) dolomite bricks (after Sittard).*

furnace hearths, where the conditions are too severe to permit of fireclay bricks being employed. They have also given service equal to that expected with straight magnesite bricks in arc furnace sub-hearths.

Semi stable bricks of the type described in Table XIV and column 1, have given excellent results in the side walls of arc furnaces and indeed proved superior in this position to both magnesite and chrome magnesite

bricks. They have, however, been largely replaced of late by metal-cased magnesite tubes, whose life may be as great as 11 weeks, compared with, say, 4-5 for semi-stable dolomite.

In some Continental plants pressed tar blocks are used both in arc furnace side walls and in open-hearth furnace backwalls (*see* fig. 94). In general, however, they are only used at plants that are already making similar products for basic bessemer converters. Side walls have been rammed from time to time with either dry or tarred doloma of particularly fine grading, but such practice has in general given way to the more convenient use of fired bricks.

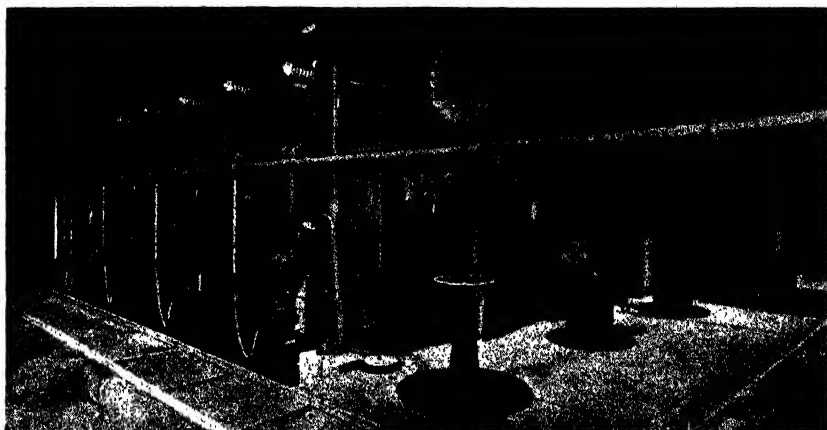
Numerous attempts have been made to use dolomite base materials for the lining of steel ladles. That such linings are more durable than the normal fireclay type has been amply proved, but their high initial price, and the numerous difficulties associated with their use has prevented their application on any large scale. The principal difficulty lies in the tendency of slag to adhere to the basic lining, thus reducing the ladle capacity. More will be said about this development in Chapter XIV on Casting Pit Refractories, but meanwhile it can be said that attempts to remove this slag usually lead to damaged bricks and consequently to a dangerous condition in the ladle.

Unfired stabilised ladle sleeves have, however, found a definite application in plants where extremely low carbon steels are made and there is consequently a very severe attack by the iron oxide rich slag on the normal fireclay type.

#### CEMENTS

Stable dolomite bricks can be set with a water mixed cement made from finely ground clinker. Semi-stable on the other hand must be set dry or with oil base cement, otherwise trouble is likely to be experienced due to hydration.

Numerous special cements based on stabilised dolomite clinker, but having various additions to control plasticity and expansion characteristics are now available for use in tap holes and for repairs to basic bottoms. They are also being used for many other purposes, *e.g.*, the production of basic linings in cupolas. The availability of such materials is already leading to marked changes in cupola practice, since longer operation becomes possible, as well as the use of limey slags to reduce the sulphur content of the metal.



*Fig. 91. Dolomite brick kiln fired with producer gas (Steelley Co. Ltd.).*



*Fig. 92. Kiln used for firing dolomite bricks showing height of setting (Steelley Co. Ltd.).*



*Fig. 93. Effect of a 4/1—Ferric oxide/lime mixture on magnesite-dolomite briquettes, after 1 hour at 1650°C. Left to right 100-0, 80-20, 60-40, 40-60 and 0-100 mixtures.*

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## Chapter V

# CHROME AND CHROME-MAGNESITE

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**C**HROME—PARTICULARLY in the form of chrome-magnesite bricks—continues to be one of the most useful and at the same time the most aggravating of all steelplant refractories. Its melting point and slag resistance are so much greater than those of silica that it might reasonably be expected to provide a solution to most open-hearth problems, including that of the all-basic furnace roof, but unfortunately only a small part of its potential value is realised because both chrome and chrome-magnesite bricks tend to spall long before they are dissolved by the furnace fluxes. Numerous explanations have been given of this annoying behaviour, *e.g.*, bursting due to solid solution of iron oxide in the chrome grains, relatively low refractoriness-under-load, after-contraction at high temperatures, and flux migration yielding a weak zone at some distance behind the face. None of these, however, explains the facts at all convincingly.

A vast amount of work has been done on the properties of chrome and chrome refractories and the constituent spinels during recent years with the object of overcoming these limitations (Special Report No. 32 of the Iron and Steel Institute contains no less than 267 pages on this subject), but this greatly extended horizon has not yet enabled the performance of chrome-magnesite bricks to be improved at all substantially. Much of this research has been concerned with the constitution of spinels and their behaviour in oxidising—reducing atmospheres and in contact with iron oxide. G. R. Rigby, who has been one of the pioneers in this field has summarised the conclusions drawn from this work in his paper on “The Spinel and Their Relation to Chrome Ores” published as part of the Jubilee Symposium on Ceramics of the British Ceramic Society, 1953—a paper heavily drawn on in the present chapter.

## THE SPINELS

It is now widely appreciated that chrome ore consists essentially of a mixed spinel phase, together with as much as 40 per cent. of gangue



material—often serpentine. The principal phase is neither ferrous chromite ( $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ ) nor the sesquioxide ( $\text{Cr}_2\text{O}_3$ ) as periodic allusions in the literature would suggest. The term spinel is used to describe a general class of minerals having the common chemical formula  $\text{R}^{\text{II}}\text{O} \cdot \text{R}^{\text{III}}\text{O}_2$ , the R term representing a range of divalent and trivalent elements respectively. To the non-technical spinel is best known as a transparent gem stone. The most familiar member of the series to the steelplant operator is magnetite ( $\text{FeO} \cdot \text{Fe}_2\text{O}_3$ ), which is formed as millscale on steel both during soaking and subsequent heat treatment. Chrome ore as mined rarely shows any obvious crystal structure, but spinels having an obvious crystallinity are readily produced by electric fusion. Thus fig. 95, page 201, shows a sample of magnesium aluminate. It would hardly be imagined that such a mineral belongs to the cubic system until it is realised that the small concentric triangles represent a section through the cube corner. Incidentally this triangular appearance is characteristic of spinels, appearing for example with magnetite (fig. 96, page 202) and as a cleavage in certain thin sections of chromite (see fig. 97, page 211).

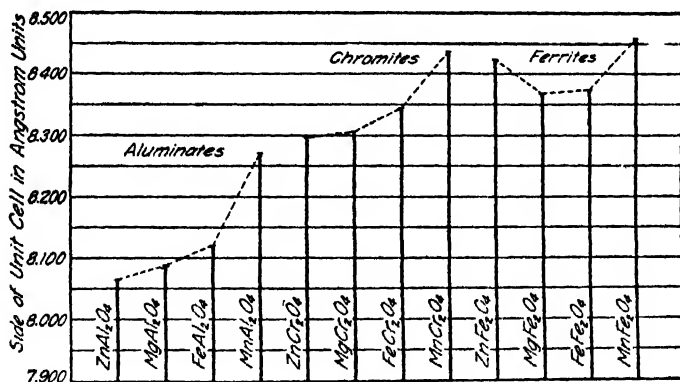


Fig. 98. Lattice dimensions of synthetic spinels (after C. W. Parmelee, A. E. Badger and G. A. Ballam).

Spinel is best identified by X-ray examination (see fig. 12, page 25), the pattern being relatively simple and only differing from one spinel to another in the displacement of the lines due to differences in fundamental lattice size. One of the first investigations of the lattice dimensions of synthetic spinels was that by Parmelee, Badger and Ballam, whose results are given diagrammatically in fig. 98. It will be seen from this that the lattice of the chromite spinels is intermediate between that of the aluminates and ferrites. With mixed spinels, such as chromite,

the lattice spacing varies *pro rata* with the content of the individual spinels present. Thus Cuban chrome ore may be considered as a solid solution of roughly equal parts of magnesium aluminate and ferrous chromite. It should be noted, however, that certain constituents, *e.g.*, iron, may be present both as divalent or trivalent ions.

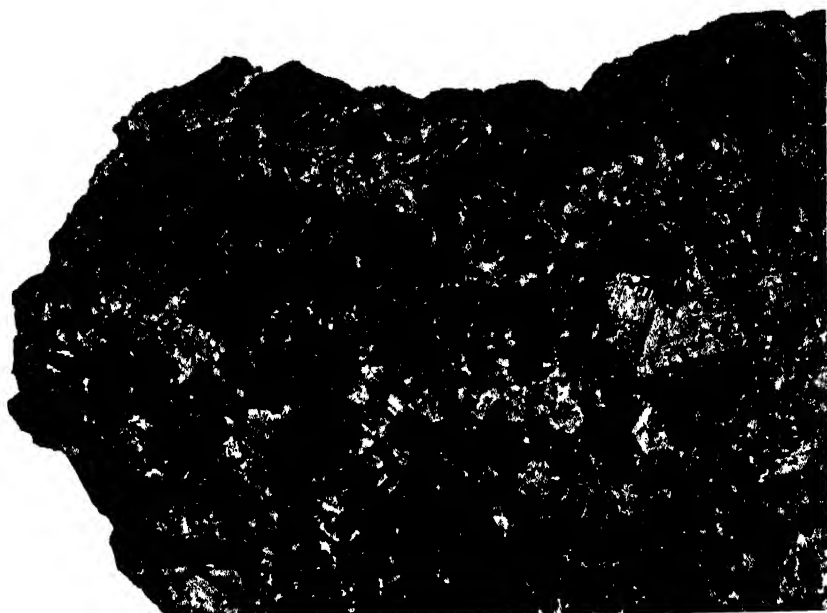
Barth and Posnjak have shown that there are really 2 types of spinels, the normal and the so-called inverse type, in which 8 of the 16 trivalent ions in a lattice group are replaced by 8 divalent ions. The general diffraction pattern remains unchanged and the different types can therefore only be differentiated by precise line intensity measurements which show up the different effect of the ions as regards their scattering power. With ordinary spinels it has not been possible to differentiate by this means, the scattering power being roughly the same, but for magnesioferrite it has been conclusively shown that the formula is  $\text{FeMgFeO}_4$  rather than  $\text{MgFe}_2\text{O}_4$ .

Certain of the spinels, *e.g.*, magnesium aluminate, can be readily synthesised by heating together the constituent oxides, the rate of combination being greatly accelerated if certain fluxes, such as boric acid (2 per cent.) are present. Chesters and Parmelee, for example, show an increase in rate of as high as 20 times with a  $\text{B}_2\text{O}_3$  content of only 2 per cent. The preparation of the ferrites is far more difficult, but Rigby gives details in "Ceramics—a Symposium" for the methods to be used. Incidentally it is now fairly certain that some of the early syntheses were not satisfactory due to lack of furnace atmosphere control. Thus Rigby and co-workers showed that when chrome oxide, ferrous oxide and boric acid were heated together, the product was not chromite but a solid solution of  $\text{Fe}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$ . They finally achieved satisfactory syntheses by using powdered iron, ferric oxide and chromic oxide heated in iron tubes. A similar technique was successfully employed for the production of hercynite ( $\text{FeAl}_2\text{O}_4$ ). Even magnetite is a difficult spinel to obtain in the pure state, because it demands strict control of oxygen pressure, but Rigby showed that when ferric oxide is heated for 2 hours at  $1400^\circ\text{C}$ . in a nitrogen atmosphere that had been freed from traces of oxygen by passing it over steel turnings the product on cooling contained 85 per cent. of magnetite. The production of mixed spinels having characteristics similar to those of chromite is even more difficult, particularly if large crystals of the type found in nature are required.

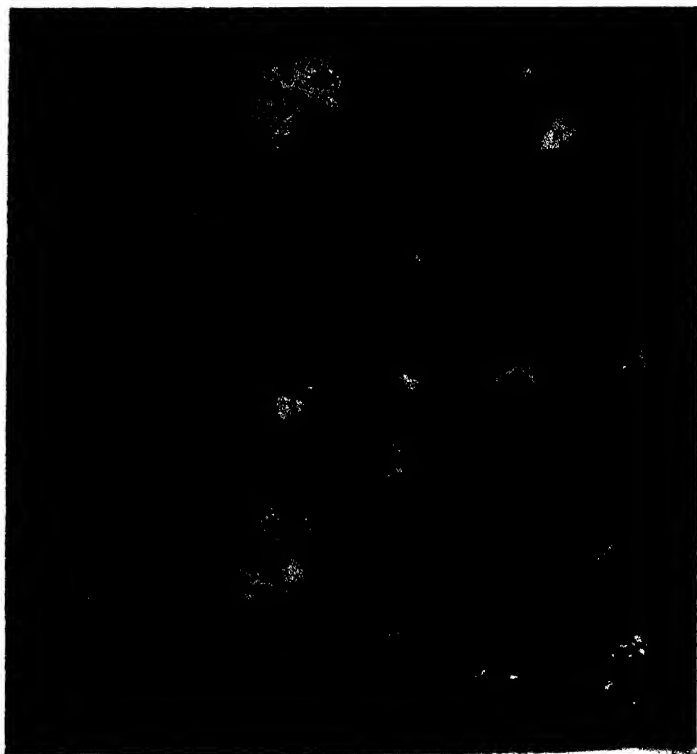
The properties of spinels have been summarised by Rigby and co-workers, some of whose data is reproduced as Table XV. In the original text many more spinels, such as nickel ferrite, cobalt stannate and the like are included. Although these cannot be classed as refractories, their

TABLE XV  
PHYSICAL PROPERTIES OF ARTIFICIAL SPINELS

<i>Name of spinel</i>	<i>Formula of spinel</i>	<i>Colour in bulk</i>	<i>Colour under microscope</i>	<i>Refractive index</i>	<i>Specific gravity</i>	<i>Cell size (Å)</i>
Hercynite	$\text{FeAl}_2\text{O}_4$	Grey-green	Pale green	1.80-1.83	4.35-4.05	8.12 8.12
Spinel	$\text{MgAl}_2\text{O}_4$	White	None	1.72	3.55	8.07 8.09
Chromite	$\text{FeCr}_2\text{O}_4$	Red-brown	Deep red	2.16	4.88-5.08	8.34 8.34
Picrochromite	$\text{MgCr}_2\text{O}_4$	Pale green	Pale green	1.90	4.40-4.43	8.31 8.31
Magnetite	$\text{FeFe}_2\text{O}_4$	Black	Opaque	—	5.14-5.2	8.37 8.39
	$\text{MgFe}_2\text{O}_4$	Red-brown	Deep red	2.34	4.20-4.49	8.36 8.37



*Fig. 95. Electrically fused spinel ( $\text{MgO}.\text{Al}_2\text{O}_3$ ) showing triangular-shaped crystals.*



*Fig. 96. Enlarged photograph of cavity in hard-fired iron ore pellet showing triangular-shaped magnetite crystals (after Ridgion, Cohen and Lang).*

behaviour, for example, in contact with iron oxide, has been of assistance in explaining the bursting of chrome-magnesite bricks. Among the noteworthy properties are the specific gravity, which is nearly twice that of, say, cristobalite, and leads in consequence to chrome-magnesite and chrome bricks being very much heavier than their silica equivalents. The optical properties of spinels have been summarised by Winchell, who prepared a square diagram with  $\text{FeO.Cr}_2\text{O}_3$  and  $\text{MgO.Al}_2\text{O}_3$  on opposite corners, to show the variation of refractive index and specific gravity with composition.

Information regarding the actual melting points of chrome refractories is scanty, but a certain amount of information is available regarding the constituent spinels. Thus magnetite has been stated to have a melting point of  $1590^\circ\text{C.}$  and magnesioferrite a melting point of  $1750^\circ\text{C.}$  Magnesium aluminate is given as  $2135^\circ\text{C.}$  and magnesium chromite as  $2200^\circ\text{C.}$  Incidentally certain of these compounds, *e.g.*, magnesioferrite, tend to change on heating, *e.g.*, they may lose oxygen before the melting point is actually reached.

Another extremely important property of certain spinels is their behaviour in alternate oxidising and reducing atmospheres. Several workers have observed that chrome bricks when given such treatment at, say,  $1400^\circ\text{C.}$ , tend to disintegrate and this may well be due to the volume changes observed for example with ferrous chromite, when alternately oxidised and reduced with hydrogen. Ferrous chromite ( $\text{FeO.Cr}_2\text{O}_3$ ) yields a solid solution of  $\text{Fe}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$  after only 2 hours in air at  $850^\circ\text{C.}$  If the product is then heated in hydrogen the solid solution is reduced back to chromite. At  $1050^\circ\text{C.}$  this second process takes about 2 hours. The reduction is accompanied by a marked growth that can be readily illustrated by making a specimen of the oxide solid solution and subjecting it to hydrogen. Even at  $750^\circ\text{C.}$  a big increase in volume occurs—of the order of 33 per cent.—corresponding to a linear movement of 10 per cent. This change is not explainable in terms of specific gravity and is now believed to be due to increasing porosity. Repeated oxidation and reduction renders such specimens extremely crumbly, and may therefore have an important bearing on the choice of chrome ores for brickmaking, where such alternate oxidising and reducing conditions are always liable to occur.

#### BINARY SYSTEMS

##### *MgO-Al<sub>2</sub>O<sub>3</sub>*

It will be seen from this system, which is reproduced as fig. 99, that the lowest melting point corresponds to an addition of approximately

## STEELPLANT REFRACTORIES

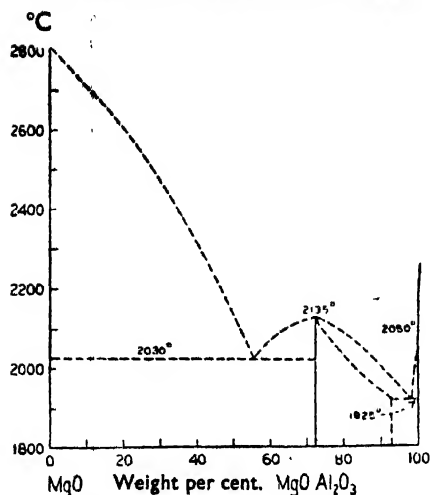


Fig. 99. System  $\text{MgO}-\text{Al}_2\text{O}_3$   
(after G. A. Rankin and H. W. Merwin).

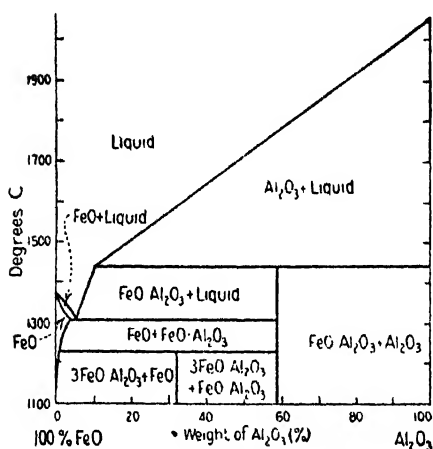


Fig. 100. System  $\text{FeO}-\text{Al}_2\text{O}_3$   
(after A. B. McIntosh, J. R. Rait and R. Hay).

2 per cent. of magnesia to alumina. Even this lowest point, however, represents a super refractory ( $1925^\circ$ ), whilst the spinel composition itself melts at  $2135^\circ\text{C}$ . As Chesters and Parmelee showed, the reaction between equimolecular amounts of magnesia and alumina results in a substantial increase in volume, a property made use of in the production of steeltight induction furnace linings. So substantial is the expansion that mixtures containing a big excess of magnesia are normally adequate. Use is also made of boric acid as a mineraliser.

$MgO-Cr_2O_3$ 

According to Wartenburg and Prophet any addition of magnesia to chrome-oxide raises its already high melting point, which for the pure material is given as 2300°C.

 $FeO-Al_2O_3$ 

McIntosh, Rait and Hay (fig. 100) showed that the addition of alumina to ferrous oxide results in a rapid drop in melting point to about 1300°C. for a 5 per cent. addition, and then a relatively steady rise to a melting point of over 2000°C. for alumina. Incidentally the corresponding spinel hercynite is periodically found as a non-metallic inclusion in steel, having presumably been formed by reaction between ferrous oxide and the alumina arising from aluminium additions.

 $FeO-Cr_2O_3$ 

This system has not been thoroughly worked out, any study of it being greatly complicated by questions of oxygen pressure—see Ternary Systems.

 $FeO-Fe_2O_3$ 

The general phase relationships in this system as worked out by White, are shown in fig. 101. Although complex the system at least

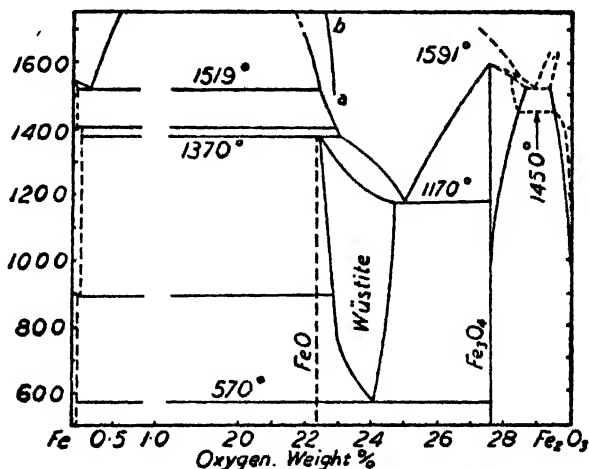


Fig. 101. System Fe-Fe<sub>2</sub>O<sub>3</sub> (after White).

makes clear one very important point, *viz.*, the unusual influence of oxygen pressure on the melting point of the oxides of iron.



## TERNARY SYSTEMS

A number of ternary systems containing spinel forming oxides have been worked out, *e.g.*, the  $\text{MgO-Al}_2\text{O}_3\text{-Cr}_2\text{O}_3$  system reported on by Wilde and Rees in 1943, and the later work of Ford and Rees (1948) on the system  $\text{CaO-MgO-Cr}_2\text{O}_3$ . Also relevant is the preliminary evaluation by Roberts and Merwin in 1931 of the system  $\text{MgO-FeO-Fe}_2\text{O}_3$ . All these systems are greatly complicated by the necessity for precise control of oxygen pressure and no attempt will be made here to reproduce the conclusions other than to say that they are characterised by various solid solutions, *e.g.*, those of  $\text{Al}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$ , and  $\text{MgO-Al}_2\text{O}_3$  and  $\text{MgO-Cr}_2\text{O}_3$ . More recently Richards and White have studied phase relations not only in the ternary systems  $\text{Fe-Cr-O}$  and  $\text{Fe-Mg-O}$  but also in the quaternary  $\text{Fe-Al-Cr-O}$  and the quinary  $\text{Fe-Al-Cr-Mg-O}$ .

## RAW MATERIALS

Since the sources of magnesite have already been discussed in Chapter III, the present section will deal solely with chrome ore.

## (a) AS MINED

The world production of chrome ore during the years 1943-1950 are summarised in Table XVI. A comparison of this with the table on p. 130 of the first edition of this book is very instructive, in that it shows that certain producers, in particular the U.S.S.R. and the Philippines, have greatly increased their output. An extraordinary feature of the distribution is that with the exception of the U.S.S.R. most of the producers are not heavy consumers, whilst most of the heavy consumers are not substantial producers. For this reason chrome ore tends to have a strongly political flavour.

Typical analyses of chrome ores from different parts of the world are given in Table XVII, which is taken from a study made by Hugill and Green. The effect of the varying composition on the properties of chrome ores is still far from clear, but it is of interest to note that certain ores, *e.g.*, those from Cuba and the Philippines run high in alumina, whilst others, *e.g.*, those from Transvaal, run high in iron oxide. All these ores contain in addition to the spinel phase, a proportion of gangue material, which ranges between 10 and 20 per cent., but may be as low as 2 or as high as 44 per cent. One of the most common constituents is serpentine (*see* fig. 102, page 212) but a wide range of other minerals has been found in chrome ores, *e.g.*, olivines, feldspars, chlorites, carbonates and quartz. The magnesium silicate ratio of the gangue material is normally rather less than 2 : 1 on a molecular basis,

TABLE XVI  
WORLD PRODUCTION OF CHROMITE  
(Metric tons)

Source	1943	1944	1945	1946	1947	1948	1949	1950
Union of South Africa ..	163,232	88,909	99,090	212,253	373,094	412,783	404,351	496,324
U.S.S.R. ..	325,000	300,000	300,000	300,000	500,000	600,000	350,000	500,000
Philippines ..	60,000	70,000	—	58,930	195,185	256,854	246,744	250,511
Cuba ..	354,152	192,131	172,626	174,350	159,209	116,624	97,368	117,358
S. Rhodesia ..	287,453	277,051	186,318	151,149	155,149	230,703	243,506	291,525
Turkey ..	154,512	182,108	146,716	103,167	102,875	285,353	434,117	350,000
New Caledonia ..	46,952	55,229	59,828	24,946	50,530	75,021	88,792	—
India ..	33,789	40,190	31,642	45,511	35,274	22,917	19,728	—
Sierra Leone ..	16,306	9,851	578	10,301	16,769	7,886	22,101	—
Greece ..	15,500	18,295	2,413	9,062	2,640	1,500	3,381	12,631
Cyprus ..	7,986	469	1,070	1,158	5,283	6,899	14,875	—
Yugoslavia ..	65,000	10,000	6,000	68,000	55,000	65,000	93,000	100,000
Bulgaria ..	5,000	5,000	—	—	—	—	—	—
Canada ..	26,848	24,543	5,221	2,821	1,961	1,556	347	—
Japan ..	58,520	71,135	28,539	7,079	2,407	9,340	27,003	31,953
U.S.A. ..	145,259	41,394	12,676	3,726	860	3,283	393	367
Albania ..	31,091	—	—	—	—	16,500	—	—
Brazil ..	7,813	4,721	1,490	174	—	1,626	—	—
Total world production ..	1,825,000	1,400,000	1,100,000	1,200,000	1,700,000	2,150,000	2,100,000	2,300,000

TABLE XVII  
CHEMICAL COMPOSITION OF RAW CHROME ORES (after Hugill & Green)

	Transvaal						Indian 1	Rhodesian 3	Shetland 2
	4	A	B	N	O	R			
SiO <sub>2</sub>	1.04	1.42	6.12	0.68	1.33	3.50	1.27	3.58	14.05
TiO <sub>2</sub>	0.74	0.85	0.56	0.95	0.84	0.87	0.37	0.10	0.10
Al <sub>2</sub> O <sub>3</sub>	16.20	12.22	18.24	15.78	17.42	15.67	12.74	13.85	12.22
FeO	20.90	25.12	20.29	18.10	20.69	23.40	7.17	11.26	9.75
Fe <sub>2</sub> O <sub>3</sub>	5.00	0.10	3.00	8.50	6.07	—	15.97	3.46	2.70
CaO	0.46	0.21	1.46	0.46	0.74	0.63	0.57	1.01	0.16
MgO	9.44	11.45	11.51	9.75	9.71	11.02	13.51	14.87	26.34
MnO	0.21	0.10	0.64	0.31	0.30	0.28	0.18	0.13	0.08
Cr <sub>2</sub> O <sub>3</sub>	45.60	47.50	36.83	45.88	42.11	43.31	45.33	47.61	27.67
CO <sub>2</sub>	0.10	0.27	0.15	0.12	0.20	0.41	0.22	1.58	0.53
H <sub>2</sub> O	0.31	0.26	1.20	0.47	0.59	0.91	2.67	2.55	6.40

	Grecian			Turkish		Cuban 6	Russian		Yugoslavian 28
	L	M	D	7	8		5	8	
SiO <sub>2</sub>	3.75	4.43	—	3.96	4.35	4.35	3.29	3.62	7.47
TiO <sub>2</sub>	0.26	0.20	—	0.33	0.30	0.30	0.33	0.32	0.30
Al <sub>2</sub> O <sub>3</sub>	22.80	23.12	—	16.40	26.80	26.80	7.62	15.96	18.94
FeO	14.01	11.94	—	8.31	8.57	8.57	4.04	1.30	11.19
Fe <sub>2</sub> O <sub>3</sub>	0.60	2.87	—	4.17	8.47	8.47	12.40	15.98	1.00
CaO	1.45	2.36	—	0.18	0.47	0.47	0.95	0.34	0.16
MgO	17.58	17.74	—	20.16	18.11	18.11	12.70	14.08	13.02
MnO	0.18	0.12	—	0.06	0.06	0.06	0.17	0.12	0.08
Cr <sub>2</sub> O <sub>3</sub>	37.41	33.96	—	45.16	30.33	30.33	56.48	46.43	43.47
CO <sub>2</sub>	0.50	1.11	—	0.18	0.22	0.22	0.58	0.52	0.68
H <sub>2</sub> O	1.46	2.15	—	1.09	2.32	2.32	1.44	1.33	3.69

and conversion of this to forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ) normally requires a magnesia addition. It has been assumed for some time that the spinel phase itself is normally balanced, *i.e.*, there are equimolecular amounts of divalent and trivalent oxides present. The position is, however, complicated by the fact that iron may occur both as  $\text{FeO}$  or as  $\text{Fe}_2\text{O}_3$ . Calculations made by Rait on the known composition of purified chrome grains suggests that if Vegard's law of a proportional change in lattice spacing with composition is obeyed then the thesis of a balanced spinel is probably justified.

It is not only the amount of gangue material present that is important, but also its distribution. As Trojer has shown, many grains in chrome ore that appear superficially to be large crystals are in fact made up of smaller aggregates sometimes separated by small amounts of gangue material. A knowledge of this fissured structure is important if the bursting characteristics of the ore in contact with iron oxide are to be predicted (*see* fig. 111, page 228).

The importance of fundamental studies on the spinels is brought out by the observation of Parmelee and Ally and later of Hugill and Green, that the thermal expansion and specific gravity of chrome ores can be largely forecast on the assumption that they consist of solid solutions of different spinels of known properties.

#### (b) PREFIRING OF THE ORE

Until comparatively recently it was assumed that when chrome ores were fired the only important change occurring was the composition of gangue material and its redistribution throughout the mass. Thus it was shown (fig. 103) that serpentine loses its water of crystallisation by about  $700^\circ\text{C}$ . and yields not a mixture of magnesia and silica but forsterite and a non-crystalline phase. More recently a great deal of work has been done on the oxidation of the spinel crystals themselves when heated. According to Konopicky and Caesar this can start at temperatures as low as  $320^\circ\text{C}$ . and is so rapid at  $1000^\circ\text{C}$ . that equilibrium is reached in about an hour. Later studies by Rigby, Lovell and Green, who took 5 chrome ores and exposed them alternately to oxidising and reducing cycles in oxygen and hydrogen, clearly showed that the  $\text{FeO}$  content could be oxidised to  $\text{Fe}_2\text{O}_3$  and subsequently reduced to its original condition. Such oxidation leads to the breakdown of ferrous chromite, yielding a solid solution of ferric oxide and chrome oxide. It is thought that the presence of these materials explains the opaque inclusions, lines, and borders, seen in fired ores, and also in some unfired chrome material. It is not surprising in view of these

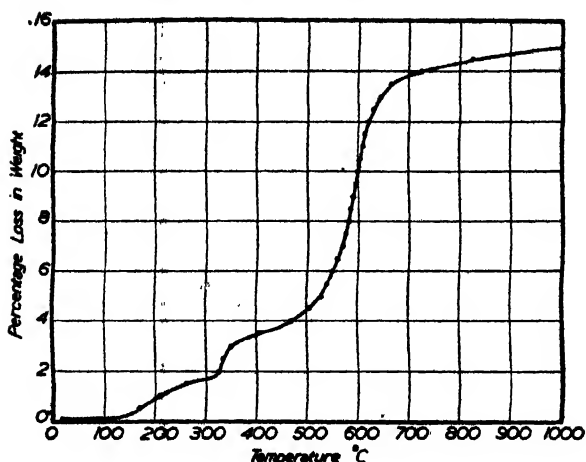


Fig. 103. Loss in weight of serpentine on heating.

observations that part of the magnesia in chrome-magnesite bricks has been shown to enter the spinel lattice. As already mentioned such alternate oxidising and reduction may well lead to a crumbly product being produced either during manufacture or service, such crumbliness being particularly common with ores initially high in iron oxide.

#### (c) OTHER PROPERTIES OF CHROME ORES

X-ray examination shows peculiar variations in crystal size and orientation of chrome ores that can be of value in identifying their origin. Studies made with some 30 samples showed that there was a marked correlation between the origin of the ore and the type of pinhole diffraction pattern obtained. Grecian chromite for example showed crystals as large as those of electrically fused magnesia and having no obvious preferred orientation. African ores and electrically

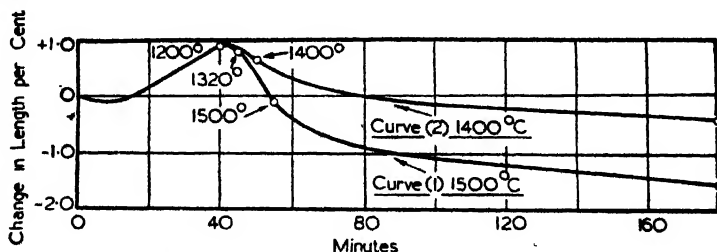
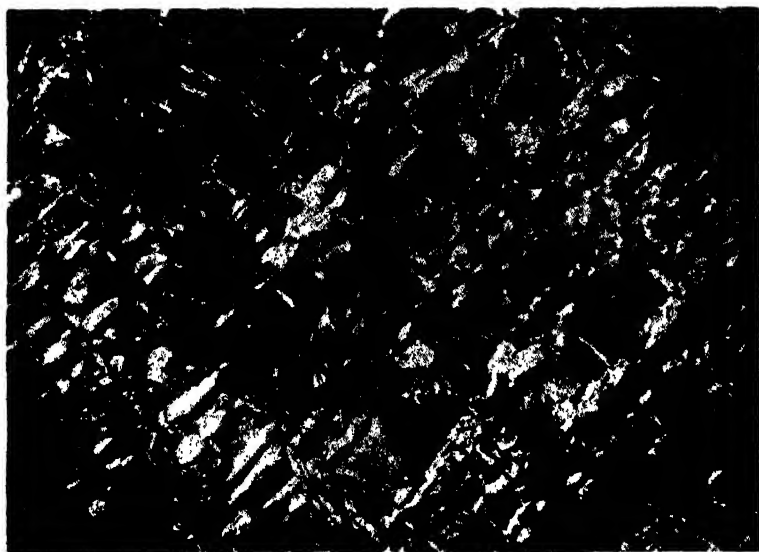


Fig. 104. Continuous firing shrinkage curves for Cuban chromite.



*Fig. 97. Photomicrograph of thin section through chrome ore showing triangular cleavage and opaque inclusions.*



*Fig. 102. Serpentine gangue (antigorite) in Shetland chromite.  
Crossed nicols  $\times 75$ .*

fused chromite also showed large crystals but not as big as those found in the Grecian chromite. Turkish, Cuban and Indian ores all showed smaller crystals with preferred orientation. A fourth group, which contained a number of African ores, showed exceedingly small crystals with little if any preferred orientation.

## THE MANUFACTURE OF CHROME AND CHROME-MAGNESITE BRICKS

### (a) CHROME BRICK

The original chrome bricks were simply made by grinding chrome ore with water and sometimes a little added bond, followed by moulding, drying and firing. They were initially used for separating basic materials, such as magnesite and dolomite, from siliceous materials, such as silica brick and fireclay brick, but later found wider application, *e.g.*, at the slag line in soaking pits. Very few such bricks are used today, it being normal to add at least 10 per cent. magnesia to the batch in order to increase the refractoriness of the gangue. This procedure not only yields a better brick but also greatly decreases the firing problem: straight chrome bricks showing a marked tendency to slump at high temperatures. Similar considerations apply to so-called "unfired chrome" bricks, where in addition to small amounts of magnesia, a chemical bond, designed for example to yield an oxy-sulphate is employed. The firing of chrome bricks affords a good illustration of the way in which the formation of the liquid matrix in combination with a refractory grog promotes shrinkage. Fig. 104 shows for example the type of curve obtained by the author when the shrinkage of chrome ore during the firing process was measured continuously. It will be seen that up to 1200°C. a typical thermal expansion occurred, followed by a shrinkage which was far greater when the furnace was kept at 1500°C. than at 1400°C. and which was still continuing when the test was stopped after 3 hours. As would be expected, such bricks show an appreciable after-contraction when refired at temperatures greater than those employed in the kiln.

Thermal expansion curves for Grecian and friable African chrome ore, reported by Heindl (*see* fig. 105) show that very substantial and quite abnormal expansions can occur with ores such as the African at quite low temperatures (900°C.), though only when they are fired under reducing conditions. In the example shown an expansion of no less than 5 per cent. linear was obtained at a temperature of only 1200°C. As Lynam, Howie and Chesters, have shown with such materials expansion



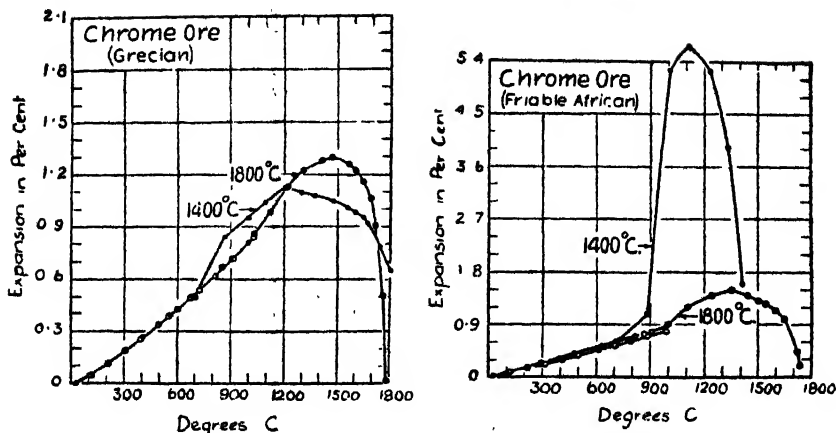


Fig. 105. Thermal expansion of Grecian and friable African chrome ore. The temperatures adjacent to the curves indicate the maximum heat treatment of the test-piece before testing. Black and white dots indicate tests made under reducing and oxidising conditions respectively (after Heindl).

in kiln firing or subsequent use leading to crumbliness and general weakness is only to be expected. A similar phenomenon has been shown by Clark and Thompson to occur when repeated changes in temperature above and below  $1380^{\circ}\text{C}$ . are employed, even though oxidising conditions are maintained throughout. Although this latter work refers to iron oxide, a similar effect may be expected in chromes rich in this material.

#### (b) CHROME-MAGNESITE BRICKS

In the early 1930's a number of workers in Germany, America and Great Britain, produced at approximately the same time, chrome-magnesite bricks of the 70-30 type, having properties much superior to those of straight chrome bricks. In particular they showed a much improved refractoriness-under-load and in general a higher resistance to thermal shock. No radical improvement has taken place since that time, the present day brick being usually a 70-30 or 60-40 chrome-magnesite mix, in which the chrome is mainly present as a coarse fraction and the magnesite mainly as a fine fraction. Such bricks may be either chemically bonded or fired in the ordinary way. They are generally made under a high moulding pressure, using either a hydraulic press or an impact type machine. The chrome ores employed vary considerably, good bricks having been made from radically different materials. In Great Britain the preference is usually for

Turkish or Grecian ore both for ease of manufacture and excellency of product.

In 1947 a preliminary attempt was made by the All-Basic Furnace Sub-Committee, a joint British Ceramic Research Association—British Iron and Steel Research Association Committee, to draw up a tentative specification for the chrome ore to be used in all-basic furnace roof bricks. It was suggested, for example, that the silica content should be between 3 to 6 per cent. and the FeO content less than 18 per cent, in order to obviate expansion in the presence of reducing conditions. The lime content was given as less than 1 per cent., so as to minimise the formation of low melting point fluxes. Chromic oxide was given as 40 to 45 per cent. Experience has shown this specification to be a satisfactory one, though doubtless equally good bricks could be made from certain compositions not falling within these rather close limits.

Both patents and publications have emphasised the marked effect of grading on thermal shock resistance. Fig. 106 shows that the highest figures tend to be obtained with batches containing approximately 60 per cent. of coarse material and not more than about 25 per cent. of an intermediate fraction. Very coarse materials also show high thermal

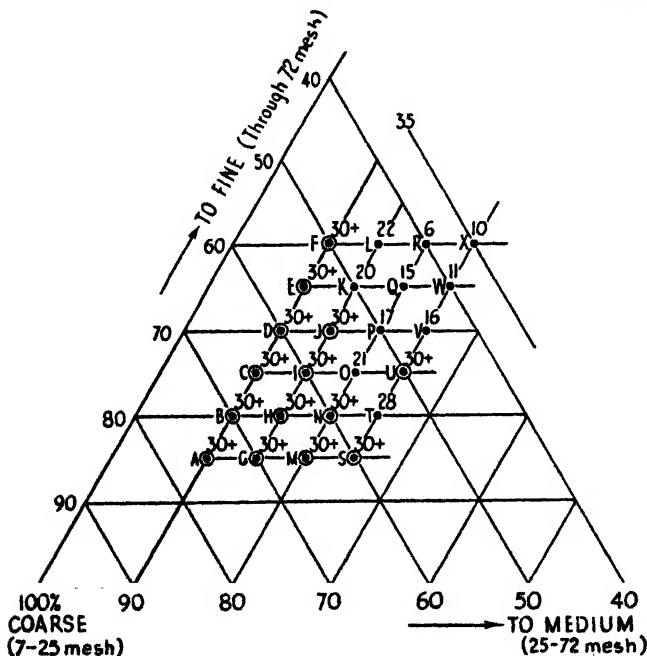


Fig. 106. Influence of grading (B.S.I. sieves) on the thermal shock resistance of 75-25 chrome-magnesite test-pieces.

shock resistance but tend to suffer from crumbliness. A popular method of achieving such gradings is the mixing of coarse chrome (on 25 B.S.I. mesh) and fine magnesite (through 72 mesh) with no intermediate material initially present—yielding a so-called gap-sized product.

No particular difficulty is experienced with the drying of chrome-magnesite bricks, since the low content of magnesia minimises the risk of trouble due to hydration. The firing treatment, however, differs greatly from plant to plant, the top temperature varying over the range 1400° to 1650°C. During recent years there has been an increasing tendency to change from coal-fired periodic to continuous type kilns fired with gas or oil. This not only facilitates obtaining such high temperatures, but also reduces troubles due to squatting by keeping the height of setting at a low figure. It also provides better control of kiln atmosphere.

Although certain properties of chrome-magnesite bricks, *e.g.*, their refractoriness-under-load, are known to improve with firing temperature, it must be admitted that good results have been obtained, for example in American all-basic furnaces and basic ends, with unfired chemically bonded bricks. This is doubtless due in part to the fact that chrome-magnesite bricks show relatively little firing expansion or contraction. Shrinkage compensation although characteristic of the 70-30 composition ceases to be found with bricks either markedly higher or lower in chrome ore (*see* fig. 107).

Although the main change occurring during the firing of chrome-magnesite bricks would appear to be the conversion to forsterite of magnesium silicates of lower  $\text{MgO}/\text{SiO}_2$  ratio, other changes do occur, as might be expected from the fundamental work carried out on spinels. Thus if electrically fused magnesia, which starts as a colourless crystalline material, is employed, it will be found to be tinted reddish-brown after firing, due to reaction with the chrome phase. Furthermore as has already been mentioned, there is a tendency with some chrome ores for the magnesia to enter the lattice of a spinel high in sesquioxides.

### (c) MAGNESITE-CHROME BRICKS

For certain purposes, *e.g.*, open-hearth furnace roofs and downtakes, magnesite-chrome bricks, *i.e.*, bricks containing less than 50 per cent. of chrome ore and more than 50 per cent. magnesite, are found to give equally good or even better service than the normal 70-30 chrome-magnesite type. The manufacture of such bricks is substantially similar to that of chrome-magnesite, the only difference being the proportions and particularly grain sizes of the two principal constituents.

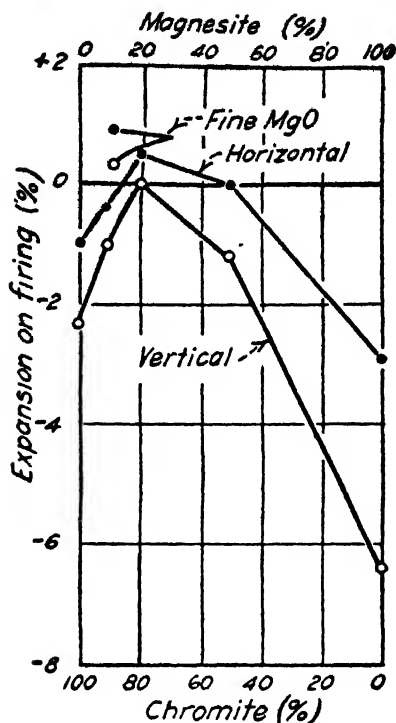


Fig. 107. Firing shrinkage (or expansion) of chrome-magnesite mixtures. Note the increased reaction with "fine MgO".

#### (d) ELECTRO-CAST BLOCKS

Electro-cast mullite has long been used for the construction of glass tank blocks, whilst a similar product made in Germany, by fusing various slags, *e.g.*, those for ferro-chrome production, has been employed in the open-hearth furnace. The Siemensit brick made by fusing together chrome ore, magnesite, and bauxite, in an arc furnace and cast into moulds, is stated to have a chemical composition as below:

$\text{Cr}_2\text{O}_3$	..	..	..	..	20-40%
$\text{Al}_2\text{O}_3$	..	..	..	..	25-45%
$\text{MgO}$	..	..	..	..	18-30%
Remainder (mainly silicates)	..	..	..	..	8-14%

Such bricks possess an extremely high refractoriness-under-load but are unfortunately rather sensitive to temperature changes.

More recently electro-cast blocks have been produced, particularly by the Corhart Company in the United States, having a magnesite-chrome composition, the principal constituents being magnesia 60 per cent., chrome oxide 20 per cent., and alumina 8 per cent. approximately. Mineralogically such bricks are similar to magnesite-chrome bricks containing a spinel phase, magnesia and forsterite, but physically they are quite different, since their porosity even including large pores is of the order of 7 per cent., whilst their crushing strength is many times that of the normal chrome-magnesite brick. Because of their low porosity, such bricks show an almost complete resistance to bursting in contact with iron oxide.

## PROPERTIES OF CHROME AND CHROME-MAGNESITE BRICKS

### (a) CHROME BRICKS

The properties of certain better type chrome bricks, as used for example in building ports in acid open-hearth furnaces, are given in Table XVIII, from which it will be seen that they are characterised by a relatively low refractoriness-under-load and thermal shock resistance. That such bricks can burst very substantially due to iron oxide absorption can readily be shown by heating up an ingot on a straight chrome brick hearth. The principal virtue of such bricks still lies in their relative inertness when placed in contact with either magnesite or silica.

### (b) CHROME-MAGNESITE BRICKS

The principal incentive behind the improvement of chrome-magnesite brick quality, has been the desire to produce a brick better than silica for use in basic open-hearth furnace roofs. The properties of a number of such bricks are summarised in Table XIX.

#### *Porosity*

It will be seen that chrome-magnesite bricks with porosities below 20 per cent. have been produced, though 22 to 26 per cent. is a more normal figure. Even with these latter porosities the resulting bricks are half as heavy again as silica, making them both more difficult to handle and demanding a stronger roof construction than would be necessary had they the same bulk density.

#### *Cold crushing strength*

Bricks having a strength of 1000 lb. per sq. in. or less are not uncommon, particularly where chrome ores high in iron oxide have been used in the manufacturing process. The use of such bricks is generally

TABLE XVIII

PROPERTIES OF CHROME BRICKS

<i>Code</i>	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
Apparent porosity, per cent. . .	22.4	28.1	28.0	26.7
Bulk density, g.p.ml. . .	3.10	2.84	—	2.93
lb. per cu. ft. . .	194	177	—	183
Apparent solid density, g.p.ml. . .	3.99	3.94	—	4.00
Permeability to air, perp. to working face, 1 skin, c.g.s. units . .	0.017	—	—	0.075
Permanent linear change on reheating, 2 hours @ 1500°C. . .	—	{ -0.22 -0.25	—	1.43
Refractoriness-under-load, 50 lb. per sq. in. °C. . .	—	—	—	—
Initial softening . .	1280	1410	1370	1350
Rapid softening . .	1350	1440	—	1440
Fail temperature . .	1370	1490	1420	1470
Thermal shock resistance, 900°C., prism test, No. of reversals . .	9	{ 8 10	—	11
Chemical analysis, per cent. . .	10	—	—	11
SiO <sub>3</sub> . .	8.52	6.4	4.00	—
Al <sub>2</sub> O <sub>3</sub> . .	11.12	16.7	19.00	—
Fe <sub>2</sub> O <sub>3</sub> . .	21.43	11.4 (FeO)	20.02	—
CaO . .	Nil	0.62	Trace	—
MgO . .	18.99	30.7	18.10	—
Cr <sub>2</sub> O <sub>3</sub> . .	—	33.1	38.58	—
Loss on ignition . .	0.17	—	0.12	—
				N.D.

objected to if only on the grounds that they suffer seriously through loss of edges and corners during handling. Weak bricks will also suffer when abraded with scrap, e.g., in the charging door jambs and arches of open-hearth furnaces. Partly for this reason they are sometimes replaced in these positions by silica bricks.

#### *Permanent linear change on reheating*

Most chrome-magnesite bricks are volume stable, or show a slight expansion on refiring at 1500°C. or even 1600°C. At still higher temperatures (*see* 2 hours at 1700°C. test), shrinkages of 2 per cent. or more are not uncommon. The question of firing expansion and, therefore, also of volume changes in subsequent use, has been explored by Lovell, who confirmed that the length changes are largely controlled by the atmosphere of the furnace.

#### *Refractoriness-under-load*

Rightly or wrongly it is normally assumed that for use in arches a brick having a relatively high refractoriness-under-load is desirable. The only escape from this conclusion could be on the basis that the bricks were suspended, or the load carried on the cold ends of the brick. Experience reported by Parnham for particular raw materials, suggests that the highest refractoriness-under-loads are obtained with 70 or 80 per cent. chrome ore mixed with 30 or 20 per cent. respectively of magnesite. Confusion has arisen in the past due to the use of the rising temperature refractoriness-under-load test, where fail points as high as 1700°C. are not uncommon with a load of 28 lb. per sq. in. The same bricks, however, maintained at 1600°C., frequently fail completely in a matter of minutes and relatively few bricks show deformation of less than 2 per cent. after a period of 1 hour.

#### *Thermal shock resistance*

It is still true that the bricks giving the best service in all-basic furnace roofs mostly show a thermal shock resistance of at least 30 reversals. Certain manufacturers have suggested that such a high thermal shock resistance is unnecessary, and by going for low porosity have produced bricks with much lower values, say 5 to 10 reversals. It is quite possible that so high a thermal shock resistance as such may not be essential, but it is more than likely that the existence of a high shock figure indicates certain other properties, notably ability to distort without fracture, that may be of considerable importance under the complex conditions of stress and strain existing in furnace roofs.

TABLE XIX  
PROPERTIES OF ALL BASIC FURNACE ROOF BRICKS

Code	A		B		C		D		E		F	
	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.
Apparent porosity, per cent.	25.4	23.6	23.9	22.6	23.0	23.8	22.7	23.5	20.7	21.5	19.6	19.1
Bulk density, g.p.ml.	2.90	2.93	2.89	2.96	2.97	2.93	3.00	2.97	3.13	3.12	3.11	3.13
Apparent solid density, g.p.ml.	3.88	3.84	3.80	3.83	3.85	3.84	3.88	3.88	3.98	3.98	3.87	3.87
Permeability to air, perp. to working face, 1 skin, c.g.s. units	0.03 0.05 0.17	0.06 0.17	0.08	0.13	0.07	0.05	0.014 0.020	0.03	0.05 0.09	0.09	0.04	0.06 0.15
Cold crushing strength, perp. to edge of brick, lb. per sq. in.	4480	3930	3000	2340	2750	2835	4200	2390	3870	2210	3990	4320
Permanent volume change on re-heating:												
2 hours 1600°C.	-1.0	-1.3	-0.7	-0.2	-2.9	-3.1	-0.8	-0.8	+0.5	No change	-0.2	No change
2 hours 1700°C.	-2.3	-4.2	-1.8	-2.2	-7.3	-5.1	-3.6	-3.6	-1.4	-1.3	-1.7	-0.7
Refractoriness-under-load, 28 lb. per sq. in. (maintained @ 1600°C. 1 hour or to fail point—5 per cent. deformation)	5% deformation from 6 to 32 min. (Inters. only)	5% deformation from 18 to 20 min. (Inters. only)	5% deformation from 18 to 20 min. (Inters. only)	5% deformation after 12 mins. (Inters. only)	5% deformation after 12 mins. (Inters. only)	5% deformation from 15 to 20 mins. (Inters. only)	5% deformation from 15 to 20 mins. (Inters. only)	5% deformation from 15 to 20 mins. (Inters. only)	3.3 to 4.3% deformation after 1 hour (Inters. only)	3.3 to 4.3% deformation after 1 hour (Inters. only)	1.4 to 4.2% deformation after 1 hour (Inters. only)	1.4 to 4.2% deformation after 1 hour (Inters. only)



TABLE XIX—continued  
PROPERTIES OF ALL BASIC FURNACE ROOF BRICKS

Code	A		B		C		D		E		F	
	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.	Rib	Inter.
Thermal shock resistance (900°C. prism test), No. of reversals .. .. .	30+	30+	30+	30+	5 to 28+	10 to 25+	4 to 30+	7 to 30+	30+	30+	30+	30+
Thermal expansion, per cent. 20° to 1000°C. .. .	—	0.71	—	0.78	—	0.87	—	0.69	—	0.76	—	—
20° to 1450°C. .. .	—	1.54	—	1.61	—	1.65	—	1.34	—	1.46	—	—
Chemical analysis, per cent.												
SiO <sub>2</sub> .. .. .	5.50	4.85	3.84	4.24	5.90	6.00	4.20	3.86	4.65	4.40	3.20	3.64
Fe <sub>2</sub> O <sub>3</sub> .. .. .	9.95	9.15	8.07	8.92	7.45	8.25	10.15	9.60	9.65	9.60	10.20	10.44
FeO .. .. .	1.00	1.40	1.70	1.70	2.85	2.60	0.65	0.58	1.20	1.60	1.37	1.44
Al <sub>2</sub> O <sub>3</sub> .. .. .	12.30	11.10	12.70	14.10	11.60	11.60	12.85	12.30	8.00	8.50	9.50	10.50
TiO <sub>2</sub> .. .. .	0.12	0.12	0.13	0.13	0.13	0.13	0.13	0.13	0.11	0.11	0.08	0.09
CaO .. .. .	1.50	1.58	1.44	1.30	2.30	2.24	1.20	1.19	0.92	0.93	1.77	1.66
MgO .. .. .	41.2	43.5	49.40	44.60	41.5	41.5	42.90	45.20	43.10	39.50	51.60	49.80
MnO .. .. .	—	—	—	—	0.17	0.17	—	—	—	—	—	—
Cr <sub>2</sub> O <sub>3</sub> .. .. .	27.6	27.8	21.90	24.10	27.6	27.4	27.8	26.9	31.70	35.30	22.10	22.50
Loss on ignition .. .	0.14	0.13	0.28	0.24	0.07	0.04	0.15	0.12	0.24	0.12	0.20	0.30

*Bursting in contact with iron oxide*

As early as 1934 Lynam and Rees reported the peculiar disintegration of chrome refractories in contact with iron oxide slags. Shortly afterwards Jay showed that the expansion was associated with solid solution of magnetite in the isomorphous chrome spinel. The amount of expansion, however, was far more than could be explained on this basis and Chesters and Lynam expressed the view that the bursting was due to iron oxide entering the brick as a liquid or vapour and dissolving in the grains to give a solid product whose volume was bigger than that of the initial volume of the chrome grains. Since the chrome grains had been pressed together in the original brickmaking, the formation of a solid skin of additional material on the surface results in their being forced apart, with consequent external expansion of the brick. Chesters likened the effect to the hydration of magnesia, where the amount of expansion occurring is far greater than that anticipated from the amount of magnesium hydroxide formed due to the upsetting of the original dense packing.

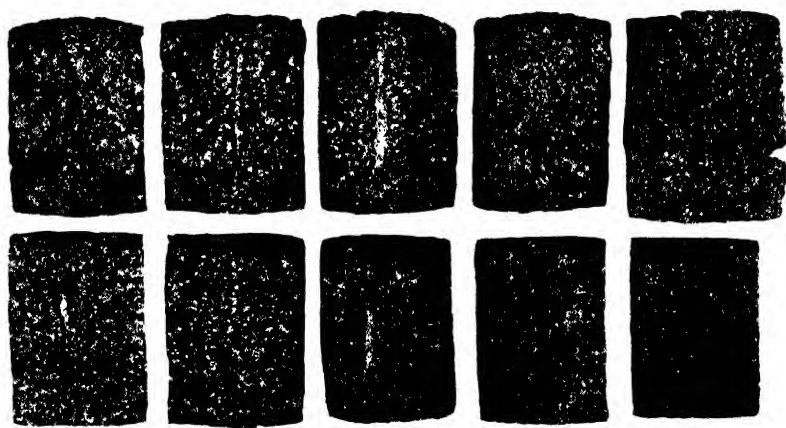
The work of Rigby has shown that the above explanation cannot be the whole story, since substantial expansion occurs when solid compacts consisting of mixtures of spinels are heated up to temperatures well below that at which any liquid phase would be present. Rigby suggests that the effect at least in the solid state is probably due to unequal diffusion rates between spinels undergoing mixed crystal formation: one crystal tending to grow at the expense of another, thus causing a change in the packing of the particles. Careful experimental data demonstrated that if spinels are classified into 4 groups, *viz.*, aluminates, chromites, ferrites, and the titanates and stannates, no growth occurs when mixed crystal formation takes place within each group. The aluminates and chromites are considered as normal spinels, whilst the ferrites, titanates and the stannates, are all inverse spinels, and growth only appears to occur when solid solution occurs between a normal and inverse spinel, *e.g.*, ferrous chromite and magnetite. This observation suggests the possibility of producing refractories of the ferrite type, *e.g.*, magnesioferrite, that would not be subject to bursting when absorbing iron oxide. Unfortunately most of these spinels have other limitations, *e.g.*, low melting point.

The original bursting test as developed by Chesters and Lynam consisted in heating up 100 g. of millscale on the 3 in.  $\times$  2 in. face on a 3 in.  $\times$  2 in.  $\times$  2 in. test piece sawn from the brick. Two such test pieces were maintained at 1600°C for 1 hour and then allowed to cool. It is generally observed that the top surface has assumed a cauliflower-like structure, and has grown substantially (*see* fig. 108,

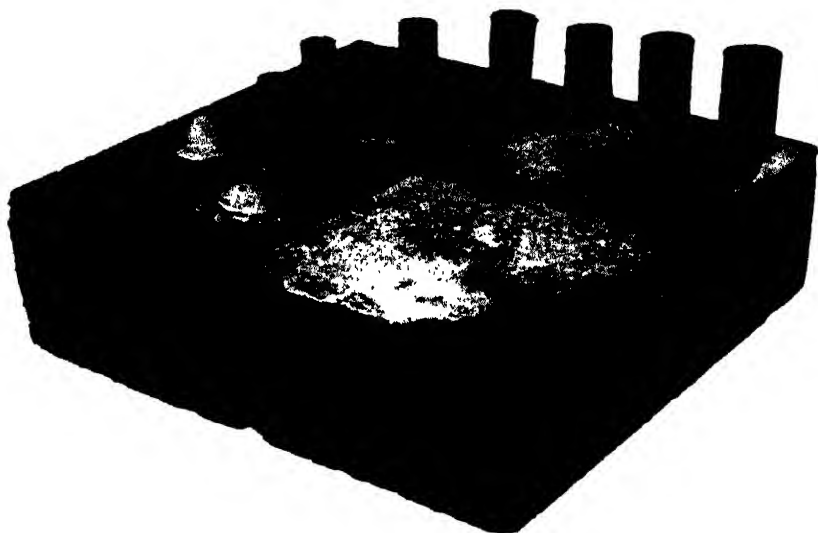
p. 225). The bursting index of the brick was defined as the sum of the percentage expansions of the three directions at right-angles, measurements being made on the top surface and for the thickness at the test piece centre. The index so obtained varies from about 70 to about 10 per cent., according to the type of brick, and is markedly lower in chrome-magnesite bricks high in magnesite or gangue. Since this was developed a modified procedure has been adopted, in which a 2 in. cube is employed, with a coating on the top surface of 40 g. of millscale bonded with sulphite lye or dextrin. The test piece is then heated to 1600° in 3 hours and then held for a further 1 hour, during which the furnace atmosphere must contain free oxygen. In this procedure the width and breadth only are measured and the results expressed as the average of the 2 percentages. Attempts to prevent bursting, *e.g.*, by inoculating the brick batch with iron oxide, have proved of little use, since the product itself is capable of absorbing large quantities of iron oxide and magnetite and continuing the expansion process. The additions of such materials as talc and serpentine greatly reduces the bursting, but generally at the expense of some other property, *e.g.*, refractoriness-under-load. The extraordinary tolerance of chrome-magnesite products for iron oxide is dramatically illustrated by fig. 109, p. 226, which shows a series of mixtures of chrome-magnesite brick with increasing percentages of iron oxide after heating to 1660°C. It will be seen that even the test piece containing 60 per cent. of added iron oxide although much shrunken has not actually melted.

Examination of used chrome-magnesite bricks, *e.g.*, from open-hearth furnace roofs shows a series of changes to have occurred. In attempting to explain these Hugill and Green prepared a special sample in the laboratory, having a rather steep iron oxide grading, and then examined a thin section cut through it. This showed quite clearly the absorption of iron oxide by the chrome grains at the working face (*see* fig. 110, p. 227), and the reaction of the silicate matrix with the incoming iron oxide, the product passing into the brick away from the working face. In practice these changes are associated with the migration of fluxes, in particular lime, from the working face, and the formation some inches behind it of a zone rich in monticellite ( $\text{CaO.MgO.SiO}_2$ ). Analyses of the various zones suggest that part of this lime is normally picked up from the furnace atmosphere.

That solid solution of magnetite occurs in the chrome of chrome-magnesite refractories subject to open-hearth furnace atmospheres is generally agreed. It is, for example, well illustrated by fig. 111, p. 228, reproduced from Trojer's 1954 paper on the degeneration of basic bricks in service. This reflected light photograph shows a large chrome



*Fig. 108. Chrome-magnesite batches of various compositions after 100 g. of mill scale has been heated for 1 hour at 1600°C. on the face shown. Unused test-piece shown for comparison.*



*Fig. 109. Right to left: 100-0, 90-10, 80-20, etc. chrome-magnesite-FeO mixtures after heating to 1660°C.*

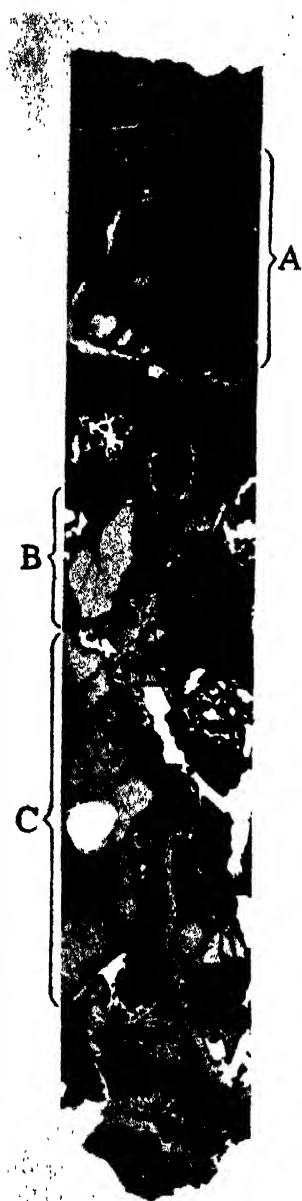


Fig. 110. Serial photomicrograph showing action of mill scale on chrome-magnesite brick ( $\times 20$ ). Infra-red radiation (after W. Hugill and A. T. Green).



*Fig. 111. Attack of iron-oxide on chrome grain in front wall brick. Bright edge indicates mixed spinel crystals and dark central region original chrome ore. Note cleavages which continue through the mixed crystal envelope. Reflected light  $\times 110$  (after Trojer).*

grain in a brick taken from the front wall of an open-hearth furnace. The light-coloured areas are an iron oxide rich solid solution of spinels formed by magnetite solution from the edges. Incidentally the fissures referred to in Trojer's 1951 paper can be seen passing through the attacked and into the unattacked area. The extent to which bursting is responsible for the loss of brick surface is however still open to doubt, certain workers giving far greater importance to thermal shock, after-shrinkage, creep, and flux migration—all combined with the stresses expected in a sprung and to a certain extent in a suspended arch. What is generally agreed is that bursting is far more serious where frequent heating and cooling of the brick surface occurs. That the rate of solid solution is only limited by the rate of arrival of iron oxide is clearly indicated by fig. 112, which shows the extraordinary speed with which solid solution can occur when millscale is allowed to run into the surface of a testpiece. It will be seen that even before the maximum temperature of the furnace was reached considerable growth had occurred, but that the rate decreased as the iron oxide became used up.

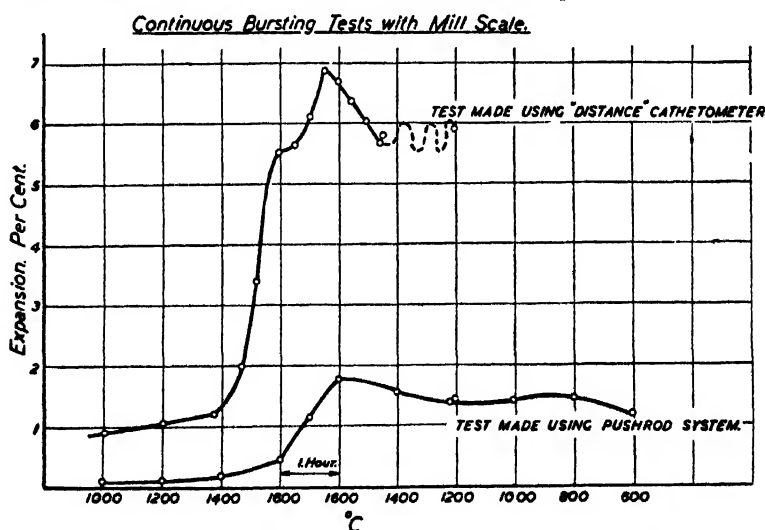


Fig. 112. Linear "bursting expansion" of chrome-magnesite brick heated in contact with mill scale. Note that expansion begins as soon as the scale melts.

#### TORSIONAL TESTS

In view of the numerous attempts made to employ chrome-magnesite bricks in the roofs of basic open-hearth furnaces, the physical properties of such bricks at high temperatures are of paramount importance. As early as 1931 Chesters and Rees showed that test pieces made from a



50-50 mixture of chrome and magnesite had a higher fail point in tension than either the chrome or the magnesite used alone. More recent work carried out at Leeds University employing torsional methods has thrown considerable light on the behaviour of chrome-magnesite bricks. Thus it will be seen from fig. 113 that even at low temperatures they do

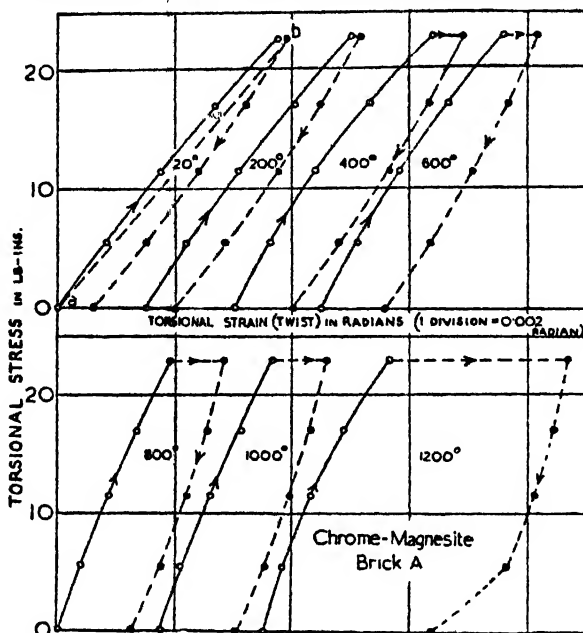


Fig. 113. Effect of applying and releasing torsional stress on chrome-magnesite test-pieces. Note the permanent set even on cold test-pieces and the increasing permanent set with temperature rise (after Roberts).

not behave elastically, *i.e.*, do not recover completely after an applied torsional stress has been removed. As the test temperature is raised the amount of permanent set increases until at 1000°C. and over it becomes quite substantial. Chung and Roberts in still later work have shown that chrome-magnesite bricks maintained under torsional loads creep quite substantially and that the creep rate goes up very remarkably once a temperature of 1300°C. is exceeded. It would seem a fair conclusion from this work that any part of a chrome-magnesite brick exposed to a load of, say, 25 lb. per sq. in. at temperatures of 1300°C. or over, will cease to carry such a load after a matter of days, the arch stresses being all thrown back on the cold ends of the bricks. The fact that the elasticity modulus is low and that even a certain amount of

permanent set can be accommodated without rupture may well be one of the reasons why chrome-magnesite bricks have been more successful in basic roofs than straight magnesite.

#### THERMAL CONDUCTIVITY

The early literature on chrome-magnesite bricks implies that their conductivity falls with rise in temperature, and that although it is higher than silica at room temperatures it is actually lower at the average temperature of open-hearth furnace roofs. Our own tests, both in the laboratory and on actual furnaces, suggest quite otherwise, the conductivities obtained at mean temperatures similar to those in an open-hearth furnace roof being higher than for silica bricks, and the external temperatures measured on new roofs appreciably higher than on their silica counterpart. Considerable further work will be required, in particular on the influence of chrome-magnesite ratio and grading on the conductivity, before any generalised statement would however be justified.

#### (c) MAGNESITE-CHROME BRICKS

Most of the properties on these bricks are so similar to those of chrome-magnesite that in the absence of chemical or X-ray analysis no differentiation would be made. Typical porosities for example lie in the 22 to 25 per cent. range. Cold crushing strength tends to run somewhat higher. Thermal shock resistances of 25 reversals and over are readily obtained, and also good volume stability and refractoriness-under-load. The main differentiation would appear to be in bursting tendency, which is normally less for magnesite-chrome than for chrome-magnesite bricks.

The above comments, however, only refer to changes over the range 70–30 to 40–60 chrome-magnesite and bricks containing much less chrome would doubtless be found to have properties closer to those of magnesite itself.

#### (d) OTHER COMPOSITE REFRACTORIES CONTAINING CHROME

Perhaps the most interesting of these is now the electrocast type, as recently manufactured for example by the Corhart Refractories Company of Louisville, Kentucky. The properties of their chrome-magnesite brick have recently been described by Hand and Baque. The batch used in such blocks would appear to consist essentially of approximately equal amounts of chrome and magnesite, and the test properties to be much what could be hoped from a brick of extremely low porosity. Thus the volume stability is excellent and the deformation after 1 hour at 1600°C. under a load of 28 lb. per sq. in. negligible. The

bursting expansion is also extremely low. The one property that might give cause for worry is the thermal shock resistance, which is of the order of 9 reversals compared with 30 + for most all-basic furnace roof bricks.

A number of other composite chrome refractories have been developed, *e.g.*, chrome-silica, chrome-alumina, chrome-dolomite and chrome-olivine. The properties of chrome-dolomite bricks have already been mentioned in Chapter IV, whilst those of chrome-silica are referred to in Chapter XIII on soaking pits.

Lynam and Rees prepared and tested out bricks made from chrome-bauxite mixtures, but came to the conclusion that they did not stand up as well under the severe conditions as did ordinary chrome-magnesite bricks. Chadeyron and Rees have since shown that the resistance of sillimanite to slags high in iron oxide can also be increased by the addition of chrome ore or chrome oxide. Bricks of the chrome-olivine type, as developed for example by Goldschmidt, may well find a use, in that they have reasonably good slag resistance, relatively low bursting tendency, and are likely to be cheaper than straight chrome-magnesite bricks due to the relatively low price of the serpentine or olivine added. Trials of such bricks in uptakes and in checker fillings have given quite encouraging results.

## APPLICATIONS

The principal uses of chrome and chrome-magnesite bricks are dealt with in Chapters VIII, IX, X, XII and XIII. Reference should also be made to the use of chrome in the form of parging, *i.e.*, patching material, for open-hearth furnace frontwalls, and the use of chrome plastics in reheating furnace and soaking pit hearths. Satisfactory cements both for jointing and for patching can be developed by the grinding up of old chrome-magnesite bricks, providing incidentally a substantial rebate on the initial high cost. The presence of iron oxide pick-up from the bath does not appear to be deleterious, whilst reasonable plasticity is developed by milling such material with water even without the addition of special bonds. Numerous proprietary cements are available for special purposes, *e.g.*, spraying through guns, and these usually contain some form of added bond, *e.g.*, clay, water glass, or ciment fondu.

One point that must never be forgotten in using chrome refractories in steel furnaces, is the possibility of chrome pick-up in the steel. Thus contamination of special casts in arc furnaces have been found to be due to the reduction of chrome bricks falling into the bath or to the use of chrome cement or chrome-magnesite bricks in the furnace taphole. For the same reason chrome is not an ideal refractory for use in induction furnace linings.



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## Chapter VI

# ALUMINO-SILICATES

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“COMMON FIREBRICKS” were for many years considered so common as scarcely to warrant any precise study. Clay in its natural form was moulded into clots, often by hand, to make probably the first, and certainly one of the cheapest, of all refractories. Silica, magnesia, dolomite, and chrome, on the other hand, were considered as rather high grade materials calling for more scientific attention. Experience shows the need to be quite otherwise. Control over the production of the newer refractories is now fairly well understood, including the changes occurring during firing, but even the nature of clay remains something of a mystery and its changes on firing the cause of many a vigorous argument.

Furthermore fireclay brick represents only part of the aluminosilicate range, which if taken to include the whole gamut from pure silica to pure alumina, provides a range of refractories with such useful and versatile properties as to be capable of dealing unaided with all the refractory requirements of an acid steel plant.

## THE PURE MATERIALS

### CLAY

Wilson defined clays as “the weathered products of silicate rocks, containing sufficient hydrosilicate of alumina in the softened condition to produce a plastic or semi-plastic mass when tempered with water”. Attempts to tighten up such definitions have led to the realisation that clays as commercially used are in fact mixtures of many minerals. At one time the breakdown was simplified into “clayite” and such impurities as mica, feldspar, quartz, and goethite. It has since been shown that “clayite” usually consists essentially of the mineral kaolinite, but that other minerals of similar composition ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) are often present, *e.g.*, nacrite, and dickite, together with very different clay minerals, such as montmorillonite and illite. That these different clay minerals are not identical can readily be shown by comparing their Debye-Scherrer X-ray photographs.

Grimshaw and Roberts have suggested a simplified procedure for the determination of mineral constitution in clays. They stress the importance of following up general examination, including the nature of the geological age and type of deposit, by pre-treatment of the clay by dispersion, sedimentation, and centrifuging. Other methods of separating minerals, *e.g.*, selective chemical action, heavy liquid separation, froth flotation, electrostatic or magnetic separation, and elutriation, may also prove necessary. Only when such a separation has been achieved can the identification of the various minerals proceed with any confidence. The techniques used for this last stage are normally chemical tests, differential thermal analysis, X-ray analysis, and microscopic examination. Given a knowledge of fundamental constitution the first really scientific step has been taken towards explaining the properties of the particular clay. Even a detailed knowledge of certain Yorkshire fireclays, as was attempted by Carr, Grimshaw and Roberts, is only of value as an example when handling clays from other sources. Thus Keller and Westcott have made a detailed study of the fireclays from the State of Missouri. Their differential thermal analysis suggests that both the flint clays and the plastic and semi-plastic clays are similar in type to kaolin, but that the high alumina clays show in addition the presence of diaspore and boehmite.

The explanation as to why such distinct minerals as kaolinite, nacrite and dickite, montmorillonite and illite, should vary so much in properties is best understood by a knowledge of their atomic structure. W. L. Bragg in his book on *The Atomic Structure of Minerals* showed that a general characteristic of clay minerals is the presence of single sheets of linked tetrahedral (Si-O) groups, similar to the double sheets present in mica type minerals, where aluminium or magnesium act as bonds. Crystals of the clay minerals are too small to be identified under the ordinary microscope, but given the far greater magnification (100,000 diameters) of the electron microscope, remarkable differences can be observed. fig. 114, p. 245, for example, shows a number of photographs reported by Humbert (Dept. of Agronomy, Ohio State University) for the clay minerals. The photographs show that the differences in crystal size and shape of the various clay minerals are at least as great as were anticipated. Indeed it seems scarcely credible that they belong to the same group. Bentonite (not shown in fig. 114) is found under the electron microscope to consist of aggregates of extremely thin plate-shaped crystals, an observation which helps considerably in explaining the high plasticity of this material due to the big surface of the adhering water films. Bentonite finds one of its biggest applications as a bond for sand in foundries, but also appears as a constituent in steelplant

refractories, *e.g.*, as a bond in sillimanite bricks. Montmorillonite and illite (a mica-like mineral) are also found as constituents in fireclays, the former being responsible at least in part for the observed fact that fireclays possess a much higher plasticity than pure kaolin, *i.e.*, china clay.

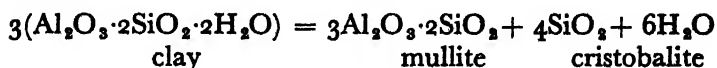
A vast amount of work has been done on the fundamental properties of clay minerals, and in particular on the way in which they are able to hold water films. E. A. Hauser, in his Edward Orton Memorial Lecture to the American Ceramic Society in 1941, summarises some of the conclusions in such work. He says for example that the ultimate clay crystal carries a negative charge either by adsorption of anions on its surface or because of an unbalanced crystal lattice. It is for this reason capable of adsorbing a number of cations to balance the charge. When the clay is suspended in water these will dissociate to a distance depending on their valency and hydration, *i.e.*, the entire clay micelle may be compared with a dissociated electrolyte. Just as sodium soap will exchange its sodium for the calcium in hard water, so each cation of the clay may be exchanged for another one. This phenomenon, known as "base exchange", although apparently far removed from the steelplant, has a very direct bearing on such important questions as the workability of cements and the density of fireclay blocks.

Those who wish to study the subject more intensively are advised to start by reading the contribution by Brindley and MacEwan of "Ceramics—a Symposium" from which fig. 115 showing the structure of kaolinite has been reproduced. The atoms in this figure are shown in approximately their correct sizes in relation to one another, though they have all been reduced about three times in relation to the lattice itself. The picture represents an enlargement of approximately 60,000,000 to 1.

#### *Changes occurring on firing*

It has long been known that clay breaks down on heating to temperatures above 500°C., the water being expelled from the crystal and an amorphous material, sometimes referred to as metakaolin, formed. To the user of refractories, however, a more important matter is the condition of the material at the end of kiln firing. In order to build up a set of standards, the late A. H. Jay X-rayed a number of clay test-pieces, ranging in type from the siliceous to the aluminous, after firing in the temperature range 900°–1400°C. His conclusions, which do not appear to have been upset by more recent work, were as follows:

- (a) Quartz in siliceous clays begins to convert to cristobalite at about 1100°C. and is not usually observable on the X-ray pattern after the 1300°C. firing.
- (b) Gamma alumina is present at temperatures between 900° and 1000°C. It is not observed at 1100°C. or over.
- (c) Cristobalite is first observed at 1100°C. It is formed, for the most part, from free silica from the dissociated clay rather than from quartz:



At higher temperatures, 1300° to 1400°C., cristobalite tends to disappear with the appearance of a diffused reflection suggesting glassy material.

- (d) Mullite is first observed at 1100°C. There does not appear to be any marked increase in the amount with increasing firing temperature. There is, however, distinct crystal development after the 1400°C. firing as indicated by the sharpness of the line pattern, especially for the high order reflections.

This is not of course the whole story, and considerable work is still in hand. Thus Comeforo, Fischer and Bradley recently combined X-ray studies with electron micrographs of the products formed on heating kaolin, and were able to show that the non-crystalline compound formed, when the water is expelled, retains the hexagonal form of the original kaolin. At a higher temperature, however, it collapses to yield crystal nuclei of mullite, which then grow in the normal way. Colegrave and Rigby, making use of thermal analysis, observed the formation of gamma alumina between 960° and 1000°C. Furthermore they showed that if a suitable flux, such as  $\text{B}_2\text{O}_3$  was present, mullite could be formed after long periods (200 hours) at temperatures as low as 900°C. They concluded that the mullite is not formed by direct combination of amorphous silica and amorphous alumina, but, at least in the presence of a mineraliser, by reaction between gamma alumina and amorphous silica. An important step forward was taken when Harvey and Birch, in collaboration with Taylor, showed that as the firing temperature rose above the point at which mullite appeared it was the size of the mullite crystals rather than their amount which increased. They decided that all the mullite that would form was already present in a specimen fired to cone 11 (1325°C).



For a long time it was thought that the needle-like crystals found in hard-fired fireclay and porcelain were the mineral sillimanite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ). It was subsequently shown, and is now accepted, that these needles are in fact the mineral mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ), so-called because it was first noted in the Island of Mull, Scotland. In ordinary fireclay bricks, the mullite crystals are too small to be identified under the microscope, but after slag attack they can grow so substantially as to be

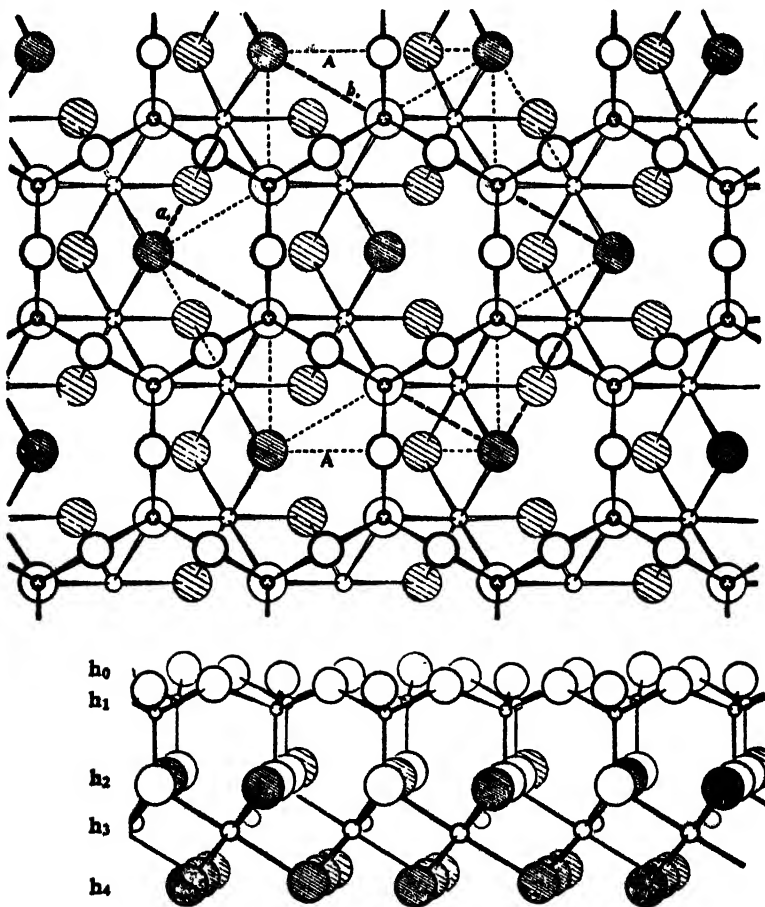


Fig. 115. The structure of kaolinite shown in plan and perspective elevation. The dotted lines outline the shapes of possible unit cells; those in bold line correspond to the actual unit cell of kaolinite. The perspective diagram represents approximately the region between the two lines marked A. Only one layer is shown; it is from the way of fitting successive layers that the triclinic symmetry arises. Scale about 60,000,000 : 1. (from G. W. Brindley and D. M. C. MacEwan).

visible even to the naked eye. Fig. 116, p. 246, shows such crystals in a fireclay crucible used for the melting of iron alloys.

#### SILLIMANITE, KYANITE AND ANDALUSITE

These 3 minerals, whose optical properties are shown in Table XX, are chemically identical ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) in their pure state, but have very different crystal habits. Sillimanite, which was the first to be used in large quantities as a refractory, is almost indistinguishable from mullite in its optical properties, whilst even the X-ray spectra are extremely similar. Given precise X-ray study, including measurements of line intensity, or alternatively the procedure recently developed by Roy and Francis, where the absorption spectra in the infra-red are compared, differentiation can however be achieved. The end product from heating all these refractories at a high temperature is mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ), the excess silica generally being present as a glass.

#### CORUNDUM ( $\text{Al}_2\text{O}_3$ )

This is formed when precipitated alumina or various aluminium salts are heated to a high temperature. It has a much higher refractive index (1.759–1.767) than sillimanite or mullite, is colourless, and usually occurs as euhedral crystals. It has a weak birefringence and extinguishes parallel to the crystal outline. Corundum is found in nature, but most of the material used for refractories is obtained by the electrical fusion of bauxite, a mineral consisting essentially of hydrated alumina. Diaspore ( $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ), which occurs for example in Missouri, is also used for making bricks with a high alumina content.

#### BINARY SYSTEM

##### $\text{Al}_2\text{O}_3$ – $\text{SiO}_2$

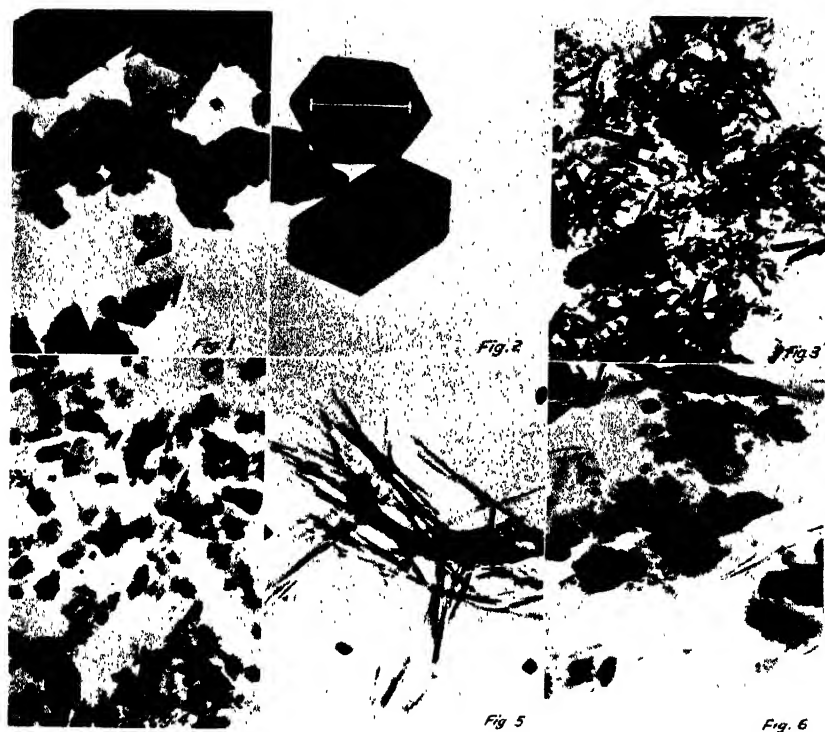
This system (fig. 117) which was first worked out by Bowen and Greig, is probably the best known and most quoted of all those dealing with refractory oxides. Even to the beginner it reveals the fact that whereas pure silica and pure alumina are both good refractories, having melting points of over 1700° and 2000°C. respectively, intermediate mixtures containing about 5 per cent. alumina melt at quite low temperatures. The effect of composition on the refractoriness in the alumina-silica system was described with remarkable clarity in Bowen and Greig's original paper as follows: "We wish to emphasise, that the 1 : 1 ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) proportion does not give the most refractory material. The  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  mixture is far superior in this respect though it is equalled by any mixture with an excess of alumina over

TABLE XX

## OPTICAL PROPERTIES OF ALUMINO-SILICATES

(Data abstracted from *Thin Section Mineralogy*, A. F. Rogers and P. F. Kerr, McGraw Hill Book Co. Inc., 1933)

	<i>Mullite</i>	<i>Andalusite</i>	<i>Sillimanite</i>	<i>Kyanite</i>
Formula ..	$3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$
Refractive indices ..	1.642-1.654	1.632-1.643	1.659-1.680	1.712-1.728
2V (axial angle) ..	45°-50°	84°	20°	82°
Optic sign ..	+	—	+	—
Colour ..	Colourless	Usually colourless	Colourless	Colourless to pale blue
Form	Long prismatic habit with nearly square cross section	Euhedral crystals or coarse columnar aggregates. Cross section nearly square	Slender prismatic crystals—felted mass of fibres	Broad elongated plates—tabular parallel to 100
Birefringence ..	Moderate. First order yellow	Moderate. First order yellow	Rather strong—up to second order blue	Moderate—up to first order red
Extinction	Parallel	Parallel in most sections	Parallel	On 100 is about 30° with length of crystals

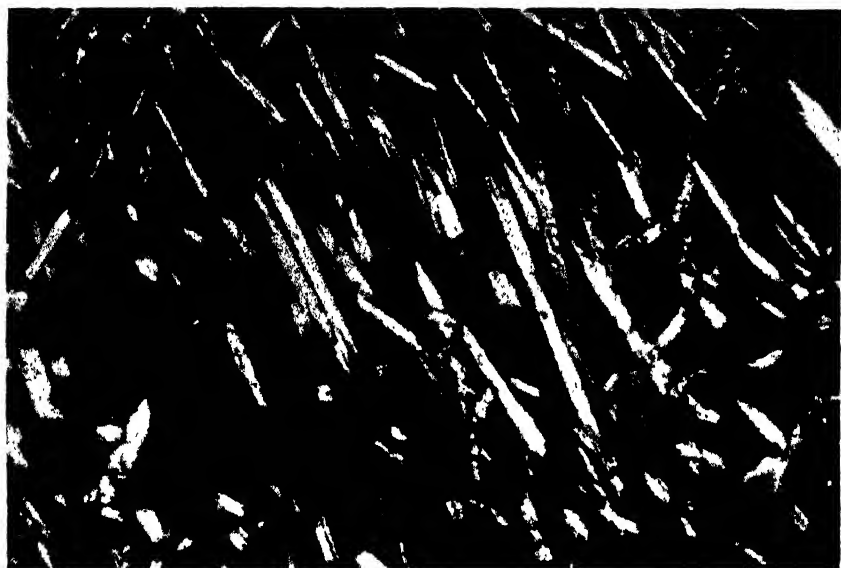


*Fig. 114. Electron microscope photographs of clay minerals.*

*Top (left to right): Kaolinite, dickite, halloysite.*

*Bottom (left to right): Sciotoxite, attapulgite, ball clay.*

*Unit of measurement (top centre)  $1\mu = 10^{-4}$  cm. (after R. P. Humbert).*



*Fig. 116. Mullite needles formed by melting iron with additions of manganese and aluminium in a fireclay crucible.*

this proportion, all of which mixtures begin to melt only at  $1810^{\circ}\text{C}$ . The slightest excess of silica over  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  is sufficient to cause a beginning of melting at  $1545^{\circ}\text{C}$ ., the amount of liquid formed at any temperature being, of course, proportional to the amount of excess silica. It is to be noted, however, that formation of some liquid at these relatively low temperatures may even be advantageous in some of the uses to which refractories are put. In the absence of other bonding material the liquid would serve as a bond which, by reason of its very high content of silica, is excessively viscous and, therefore, does not lead to ready deformation of the mass. Occurring as it does as the matrix of

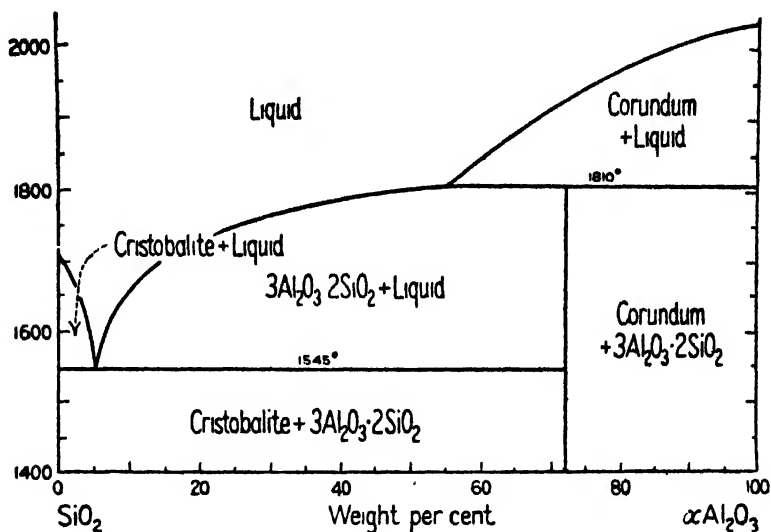


Fig. 117. System  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  (after N. L. Bowen and J. W. Greig).

interlocking needle crystals of  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ , the material as a whole is quite strong unless the temperature is raised to such a point that the liquid is present in considerable amounts, which temperature depends of course on the total composition and is highest for those compositions which approach  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ . Moreover, the interstitial liquid ordinarily does not recrystallise on cooling but forms a glass which, since it is so highly siliceous, tends to endow the whole mass with the desirable characteristics of silica glass, principally resistance to fracture upon sudden heating and cooling. These remarks apply with special force to the pure alumina-silica mixtures. Where small amounts of alkalis, lime, magnesia, etc., are present all of these enter into the liquid

which is very much increased in amount and loses much of its excellence as a glass because not so siliceous. Broadly speaking the refractory power of alumina-silica mixtures increases directly with the alumina content, omitting, of course, those mixtures with more than 95 per cent. silica. There is no maximum melting point anywhere in the series. To be sure all mixtures between  $\text{SiO}_2$  and the compound (mullite) begin to melt at the same temperature but the amount of liquid formed at any temperature decreases as the alumina content increases and becomes vanishingly small as the compound is approached, so the above generalisation remains substantially true."

It should not, however, be assumed that aluminosilicate refractories behave as equilibrium products, since a brick composition containing, say, 30 per cent. alumina, may contain a lot of free quartz even after firing and in addition substantial quantities of other oxides, such as iron oxide, titania and the alkalis. Numerous examples could be given of non-equilibrium conditions, but one will suffice to illustrate the problem: in research on acid Bessemer tuyere life, it was found that clay of approximately 40 per cent. alumina content was being let down by the addition of 10 per cent. of raw quartzite. Although according to the equilibrium diagram this would result in a product having a lower melting point, such tuyeres were found to have an improved life. Examination of slagged tuyeres and of the slag itself, showed that the added quartz in the batch did not have time to react with the fireclay either in the kiln or in use, and the product was effectively a high-grade fireclay brick studded with small silica inserts, which showed a remarkably high resistance to attack by iron-rich slags.

#### TERNARY SYSTEMS

##### $\text{FeO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

Thanks to the works of Snow and McCaughey, and later of Schairer, a fairly complete picture exists (fig. 118) of this system. Even a casual examination shows that the addition of ferrous oxide to a brick of 30 per cent. alumina composition would be expected to result in a rapid fall in melting point, to a point which even with pure oxides would hardly exceed  $1000^\circ\text{C}$ . Furthermore such low melting points could be expected even with relatively aluminous refractories, an observation in line with the observed attack of millscale on fireclay or even sillimanite hearths at temperatures of  $1200^\circ\text{C}$ . upwards. Several binary compounds occur in this system apart from mullite, *e.g.*, hercynite ( $\text{FeO}\cdot\text{Al}_2\text{O}_3$ ) a spinel, and fayalite ( $2\text{FeO}\cdot\text{SiO}_2$ ) but strangely enough there is no evidence of any ternary compounds being formed.

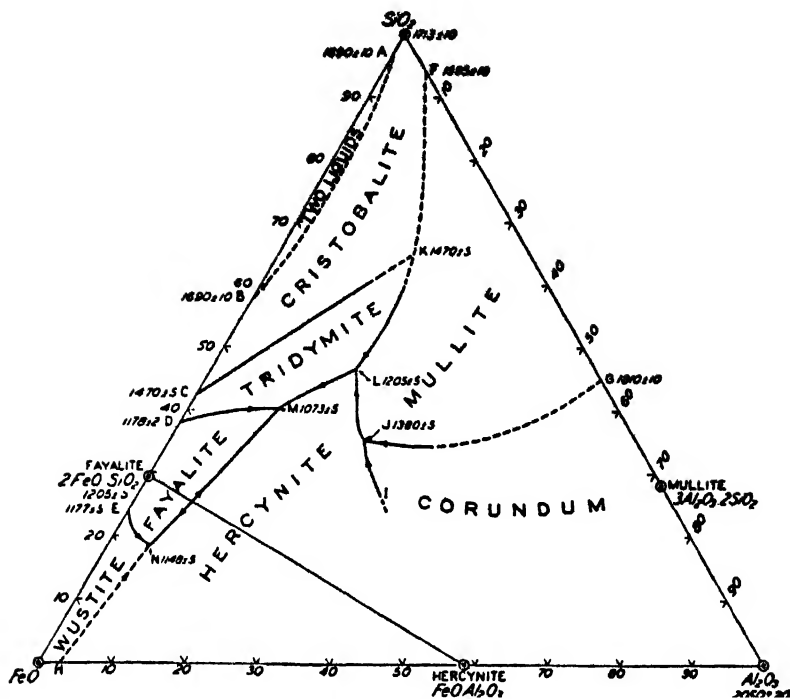


Fig. 118. System  $\text{FeO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  (after Schairer).

### $\text{MnO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

This system (fig. 119) is in many ways similar to the previous one, showing a low melting point region in a similar part of the diagram. Two ternary compounds  $3\text{MnO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$  (spessartite) and  $2\text{MnO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$ , have, however, been found. The first is a member of the garnet family, being already known in nature, but the second does not appear to have been recorded except in synthetic melts. As with the  $\text{FeO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  system, the resistance to attack by  $\text{MnO}$  increases with the alumina content of the mix, but not at any very remarkable rate. It is for this reason that sillimanite, although undoubtedly better than fireclay in resistance to attack by iron and manganese rich slags, is very rarely an economic proposition, any improvement in performance being more than offset by increased price. With very high alumina compositions (over 70 per cent.) bricks markedly more resistant to iron oxide attack are attained. Such bricks are for example used in the hearths of large slab reheating furnaces.



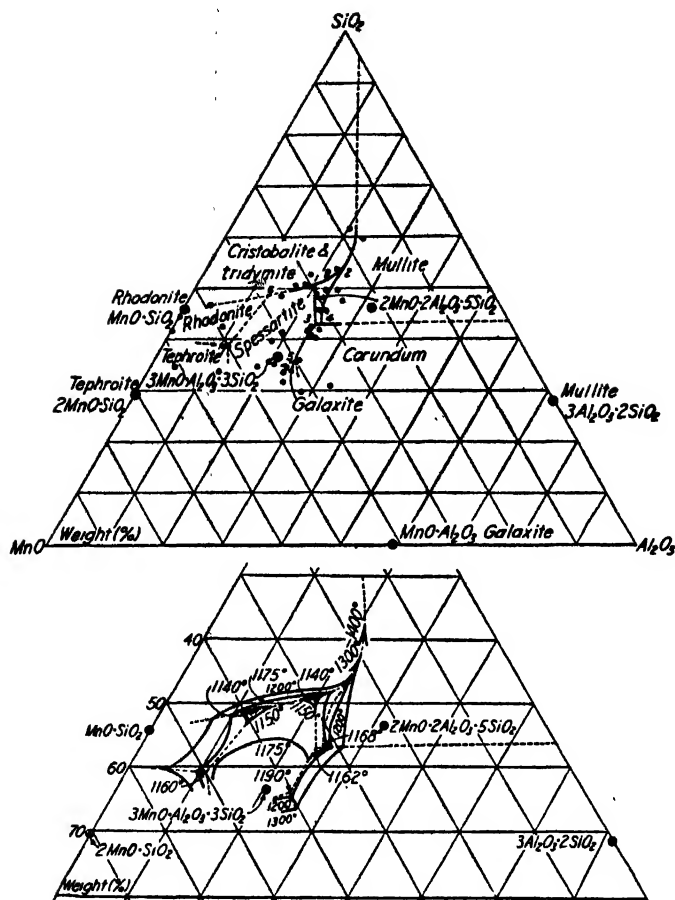


Fig. 119. System  $\text{MnO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  (after R. B. Snow).

### $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

This system (fig. 120) is of more interest to ironworks than steelplant operators, being the basic diagram for blast-furnace slags. It enables little to be deduced regarding the attack by lime rich steel slags, such as those found in the basic open-hearth, since these rarely exceed 50 per cent. lime, and other phases, notably  $\text{FeO}$ ,  $\text{MnO}$  and  $\text{P}_2\text{O}_5$ , are present in substantial quantities.

### $\text{MgO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

The melting points in this system (fig. 121) are of interest to users of refractories in the pottery industry, since it has been shown that the

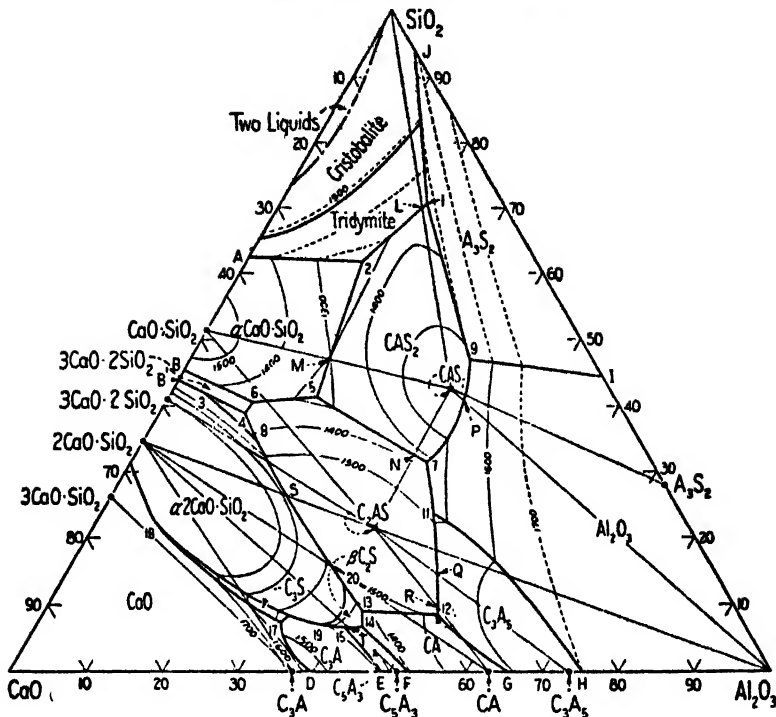


Fig. 120. System  $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  (after Greig, Rankin and Wright).

formation of cordierite ( $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$ ) in saggars, results in a marked improvement in thermal shock resistance, presumably due to the very low coefficient of expansion of this mineral. It may someday achieve a similar use in, say, casting-pit refractories, though it should be noted that any substantial addition of magnesia is likely to result in considerable fall in refractoriness. The system also shows the danger of heating fireclay bricks in contact with magnesite bricks.

A great deal more work on equilibrium systems is required before the melting point and phase relationships of alumina-silica refractories in contact with other oxides can be predicted. Particularly useful would be the complete evaluation of the system  $\text{FeO}-\text{MnO}-\text{Al}_2\text{O}_3-\text{SiO}_2$ , and further of the quinary system containing these oxides together with  $\text{CaO}$ .

## RAW MATERIALS \*

### FIRECLAY

Great Britain is fortunate, in that it has numerous deposits of fireclay distributed throughout the country. The most refractory clays are those

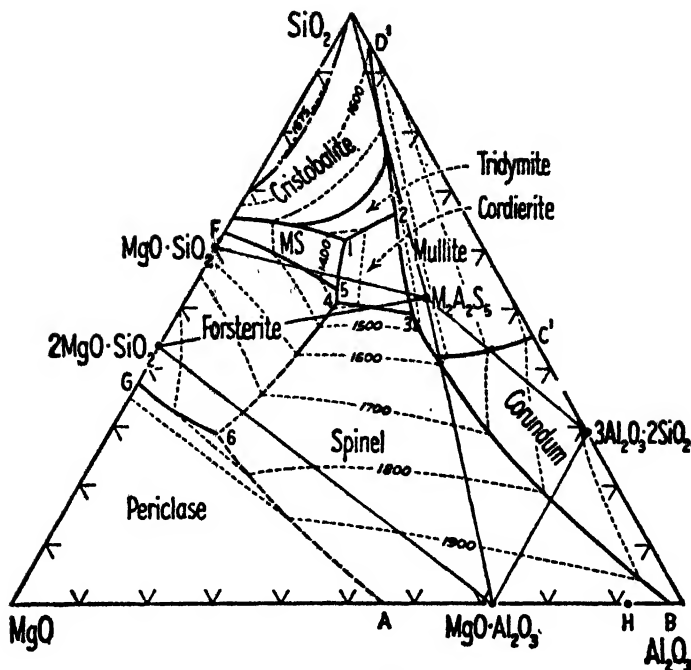


Fig. 121. System  $MgO-Al_2O_3-SiO_2$  (after Greig, Rankin and Merwin).

found in Scotland, which, together with china clay, form the basis for the first quality and special fireclay bricks. Limited deposits are also available in Cumberland with a refractoriness as high as  $1730^{\circ}C$ . According to British definitions, B.S.I. 1902:1952, a firebrick is a brick that in the fired state consists essentially of aluminosilicates and silica and shows on analysis less than 78 per cent. of silica and less than 38 per cent. of alumina. An aluminous firebrick on the other hand is one that contains between 38 and 45 per cent. alumina, the remainder being essentially silica. Bricks containing at least 78 per cent. but less than 92 per cent. of silica are referred to as siliceous firebricks.

American definitions are on a radically different basis. In A.S.T.M. designation C27-41, four classes of fireclay are recognised. *viz.*, super-duty fireclay brick, defined as having a pyrometric cone equivalent not lower than cone 33 on the fired product, not more than 1 per cent. linear shrinkage in the reheat test ( $2910^{\circ}F$ ,  $1600^{\circ}C$ ), and not more than 4 per cent. loss in the panel spalling test. High heat duty bricks may conform to one of the following requirements; pyrometric cone equivalent not lower than cone 31-32, or not more than 1.5 per cent.

deformation in the 2460°F. (1350°C.) load test. Intermediate duty fireclay bricks should either have a cone equivalent not lower than cone 29, or show not more than 3 per cent. deformation in the 2460°F. (1350°C.) load test; whilst low duty fireclay bricks are stated to have a pyrometric cone equivalent not lower than cone 19.

Until recently relatively few bricks were produced in Great Britain in the range between the 42 per cent.  $\text{Al}_2\text{O}_3$  fireclay brick and the 60 to 70 per cent.  $\text{Al}_2\text{O}_3$  sillimanite type refractories. Several bricks are, however, now available, *e.g.*, having an alumina content of 50 per cent. some of which are made from South African andalusite. In America the range is covered far more comprehensively, thanks to the availability of diaspore clays, such as are found for example in Missouri.

British users are frequently confused by the term flint fireclay, described in A.S.T.M. designation C71-51, as a hard or flint-like fireclay, occurring as an unstratified massive rock, practically devoid of natural plasticity and showing a conchoidal fracture. The existence of these clays in the United States has enabled a range of bricks to be produced in which the flint clay takes the place of what in British brick would be pre-fired grog. In 1952 L. Halm reported a comparative study of American and French flint clays, and showed their remarkable similarity by chemical analysis, microscopic examination, and thermal analysis methods. The range studied showed an alumina content on the unfired clay (ignition loss 13 to 14 per cent.) or 38 to 39 per cent., other impurities being at a relatively low level. The pure white varieties were found to have a pyrometric cone equivalent of Seger cone 34-35 (1750° to 1770°C.) which fell to cone 33 (1730°C.) on some of the less pure samples.

Although the initial selection of refractory clays is usually made on the basis of chemical analysis or refractoriness, other properties, *e.g.*, plasticity, drying shrinkage, firing shrinkage, and after contraction, may prove equally important. Bricks are rarely made from one raw clay, the desired properties generally being obtained either by the use of grog, or by blending of one clay with another. Thus many American bricks are prepared by bonding flint clays with plastic clays. The amount of grog, which is generally ground up wasters but may in certain cases be specially prepared, *e.g.*, by firing raw clay in a rotary kiln, varies from nil to over 90 per cent. in the Scheidhauer and Giessing process. The properties of the brick can vary greatly according to the amount and size of the grog present and the final firing temperature, both of which are adjusted according to the purpose for which the brick is required.

The principal impurities in fireclay bricks are silica (as raw quartz) and iron oxide, only a few per cent. of the latter being sufficient to lower the melting point by 20° to 30°C. The other impurities, *e.g.*, titanium oxide, lime and magnesia, have less effect, but alkalis if present to the extent of more than 1 to 2 per cent. may cause serious vitrification and lead to shrinkage and spalling in service. As will be seen later, organic constituents may also be present and lead to considerable trouble when not fully oxidised in the early stages of kiln firing.

#### SILLIMANITE, KYANITE AND ANDALUSITE

In the period between the World Wars, considerable quantities of sillimanite were imported, *e.g.*, from the Khasi Hills in Assam and from India. Although these deposits were relatively inaccessible and undeveloped, substantial quantities were employed in Great Britain, *e.g.*, for the production of glass tank blocks. During and after the Second World War, such sillimanite has been relatively unobtainable and in consequence bricks of this type have been made from kyanite, and more recently andalusite from Africa. These latter minerals are also found in commercial quantities in the United States, *e.g.*, in North Carolina and California. The development of these refractories, and the possible use of topaz as an alternative, have been fully discussed in an article by McVay and Wilson. Since certain of these alternatives to sillimanite are found in very much larger quantities and some of them contain even a higher content of alumina, the production of even better refractories of this type should ultimately prove possible.

#### CORUNDUM

Although a certain amount of actual corundum is mined, most of the alumina for really high alumina bricks comes from the calcination or electrical fusion of the hydrated forms, *e.g.*, bauxite and diaspore. Of these bauxite has the wider distribution, the principal deposits being in Russia, France, the United States, Dutch Guiana, British Guiana, Hungary, Italy and Yugoslavia. Minor quantities are found in other countries, *e.g.*, in Greece and Northern Ireland.

### MANUFACTURE OF ALUMINO-SILICATE BRICKS

#### FIRECLAY

Most present day fireclay bricks are manufactured either by dry pressing or by extruding a column of clay, followed by repressing to give the desired shape. Limited quantities of clay are still worked by hand,

using the plastic process, but in general the use of this process is limited to less modern factories or to the production of small quantities of special shapes. In the dry-press process the clay batch is mixed with a relatively small amount of water (5 to 8 per cent.) poured into press boxes and moulded by means of mechanically or hydraulically applied pressure. With some machines a vacuum is applied to the press box to reduce the risk of lamination and cracking due to trapped air. In the second mentioned process the clay passes through a pug mill, where it is mixed with a rather greater amount of water (10 to 15 per cent.) and is then extruded through an auger machine, often under a partial vacuum, cut into lumps of the desired size, and then repressed. This latter process is particularly useful, *e.g.*, for the production of hollow-ware for use in the casting-pit.

The drying and firing procedures vary considerably from one plant to another. Large numbers of shapes are still dried on wooden pallets, placed on steam heated floors, but in the more modern works tunnel driers for controlled humidity and temperature are used. Kilns can be divided into two main types, the periodic and the tunnel. In the former the bricks are often stacked to very substantial heights, subjected to the firing schedule and allowed to cool; whilst in the tunnel kiln they are placed on special cars which are pushed through an initial drying and heating-up zone and thence through a firing and cooling zone. The latter procedure has the advantage of greater fuel economy and tighter control over firing and cooling schedules, but lacks the flexibility of the small kiln, whose schedule can be suited to even a small quantity of goods requiring to be fired. Although the general procedure in making firebricks is much the same for all types, numerous modifications are introduced, according to the type of material ultimately required. Thus different clays may be blended and both the grog addition and the firing treatment varied. If bricks are required with a very high slag resistance but thermal shock resistance is not particularly important, a finely ground batch may be used with only a small grog addition and de-airing and hard firing employed to give a brick of low porosity. The use of such a brick for certain other purposes, *e.g.*, reheating furnace roofs and side walls, may, however, prove disastrous, due to thermal shock spalling. For such purposes bricks having a rather higher porosity and grog content, and consequently a greater flexibility both when cold and at temperature, may be preferable.

German firebricks have normally contained a rather high proportion of grog. During the last 20 or 30 years, a process capable of working with 90 per cent. and over has been developed by Scheidhauer and Giessing, A. G., who showed that if the surface of the grog grains was

covered with a deflocculated bonding clay slip, then a mixture of dry pressed consistency was produced that could be made to bond together very strongly by pneumatic tamping in cast-iron frame moulds. The water content of such blocks is only of the order of 5 per cent. and drying times of a day rather than a month become possible. Furthermore the firing shrinkage is low, as is also the final porosity, figures of less than 20 per cent. being common. In general bricks having a high grog content tend to be rather weak, but bricks made by this process are stated to have cold crushing strengths of the order of 10,000 lb. per sq. in.

The finishing temperature for firing fireclay bricks varies greatly, but generally lies in the range between 1100° and 1400°C. It is lowest for relatively non-refractory clays, such as those used in the casting-pit, and much higher for the 42 per cent. alumina type employed for example in open-hearth furnace checker chambers and settings. During recent years considerable progress has been made in the United States by the so-called super-duty brick, whose alumina content usually lies in the 43 to 45 per cent. range. Fired at a suitably high temperature such bricks have a relatively low porosity and consequently a very high slag resistance. They are, therefore, peculiarly useful for such positions as the lining of hot metal mixers. In addition they possess considerable volume stability. Although such bricks were originally made from diaspore type material, there is now an increasing tendency to use bauxitic clays, and it is probable that the manufacture of brick from these clays will become more common outside the United States.

An intermediate type brick, though of rather lower alumina content than the above super-duty, is that made from china clay. Although very low in iron, this material tends to be high in alkali, and, therefore, has a considerable firing shrinkage, which necessitates its precalcination where it is to be employed in substantial quantities. The production of such grog can conveniently be done in rotary kilns, a method also employed for the production of fireclay grog in the United States, in Italy and elsewhere.

Although it is the final soaking period that is generally of the greatest importance to the steelplant operator, there is another stage in firing which may prove vital if bricks of higher slag resistance are to be obtained. This is the so-called oxidation period, mainly below 1000°C., when carbonaceous material is burned out. If the firing is too rapid during this process, then "black-hearting" or "coring" may result. For certain applications, *e.g.*, steel ladles, the presence of such black-hearts is considered highly dangerous, since they are liable to eat out very rapidly as soon as the metal reaches them. Cases are known

for example where a breakout has occurred by one or more such bricks being cut through in a single heat. The factors controlling such coring of fireclay refractories have recently been studied by Cowling and Elliot, who showed it to be due to incomplete removal of carbon up to a temperature of  $900^{\circ}\text{C}$ ., subsequent removal being made difficult either by contraction of the body or by the lack of sufficiently strong oxidising conditions. They found it was frequently associated with the use of too little grog or excessive moisture in the ware when set.

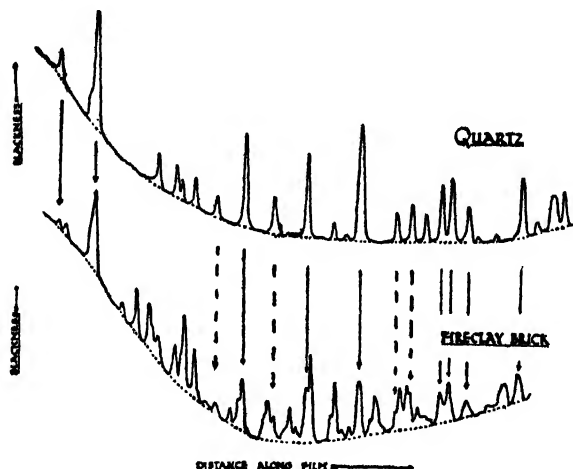


Fig. 122. Photometer curve of X-ray (Debye-Scherrer) photograph of siliceous fireclay brick showing presence of quartz.

With high alumina bricks, by far the greatest risk is under-firing, a 42 per cent. alumina brick when fired beside, say, a 35 per cent. alumina brick, being relatively soft and porous after heat treatments that caused the latter to be over-fired and even glassy. It is for this reason usual to separate bricks of different alumina contents, so as to enable different firing treatments to be given. In some plants, however, a compromise is made, the more refractory material being placed on the top of the setting.

X-ray treatment provides a ready means of describing the type of product, and in certain cases enables an estimate to be made of the firing treatment. Thus figs. 122, 123 and 124, show photometer curves for siliceous fireclay brick, and for a medium alumina brick after normal and particularly hard-firing. It is interesting to note that in the first case there is considerable residual quartz after firing, showing that equilibrium has not been reached. The medium alumina brick shows no



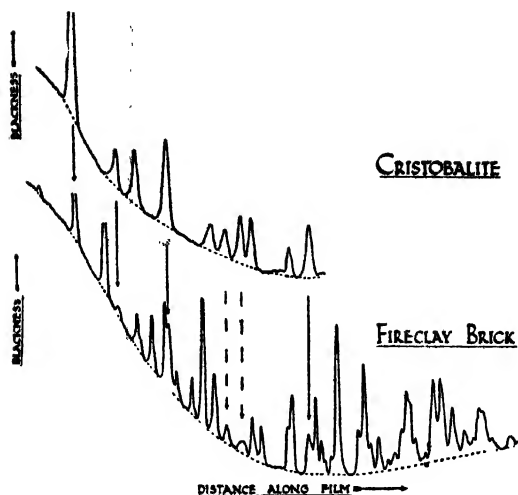


Fig. 123. Photometer curve of X-ray (Debye-Scherrer) photograph of medium alumina fireclay brick after normal firing, showing presence of cristobalite formed by breakdown of the clay.

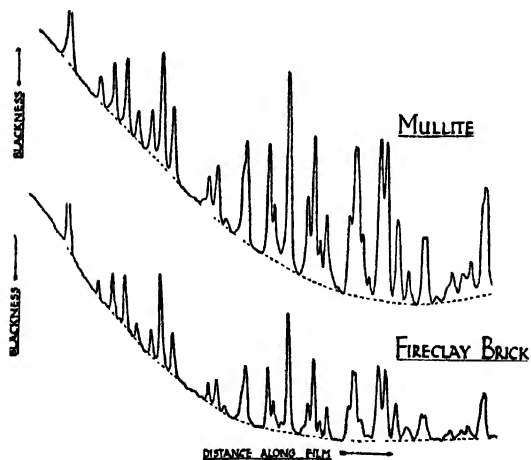


Fig. 124. Photometer curve of X-ray (Debye-Scherrer) photograph of hard-fired medium alumina fireclay brick showing mullite as the only crystalline phase.

free quartz, but does show the presence of cristobalite, part of which presumably came from the silica from the clay. Hard-fired bricks, particularly if they contain appreciable alkalis, show the lines of mullite only, the remainder of the brick consisting of a siliceous glass.

That X-ray examination can be used to estimate firing temperature was effectively shown by Jay, who compared various bricks with standard testpieces of similar clay fired to known temperature. Jay was able in this way to state that a brick normally fired to  $1200^{\circ}\text{C}$ . had been on a particular occasion fired to nearer  $1400^{\circ}\text{C}$ . and was, therefore, able to explain to the satisfaction of all concerned, the low thermal shock resistance of a particular delivery. The technique employed by Jay is described in more detail in the chapter on casting-pit refractories, under the heading of "Ladle Sleeves". More recently Richardson and Wild have carried out a systematic examination of the effect of firing on china clays, ball clays, Scottish fireclay, Stourbridge fireclay, and halloysite. Their conclusions in no way invalidate those of Jay, though they do emphasise the necessity of having standard photographs for the clay under consideration, and the danger of using the behaviour of one clay to produce information regarding the firing of a brick made from another material. Given such standards they were able to show that two samples of a particular brick had been fired at approximately  $1000^{\circ}$  and  $1150^{\circ}\text{C}$ ., whilst other tests on the same product indicated temperatures of  $980^{\circ}$  and  $1100^{\circ}\text{C}$ . respectively.

An understanding of the changes undergone by alumino-silicate compositions on firing is made easier by a study of fig. 125, which shows the results obtained by Navratil and Fessler, on the changes occurring when porcelain batches were fired at  $1400^{\circ}\text{C}$ . Porcelain itself is essentially a mixture of mullite and glass, and is normally formed by heating bodies made from a mixture of clay, quartz, and felspar. The ordinary firebrick does not contain enough felspar to form porcelain, but prolonged heating, or contact with alkalis, such as may occur for example in a blast-furnace stove, does result in the formation of porcelain-like material. Where only the working face is transformed considerable shrinkage may occur, and the resulting stresses lead to spalling away of the surface layer or piece. Fig. 125, is particularly interesting, because it shows that the ease with which porcelain type structures are produced decreases as the silica content increases. This may explain why some siliceous fireclay bricks show less tendency to shrink and become "potty" under alkali attack than bricks of, say, 40 per cent. alumina content. It also shows the danger of having a high initial alkali content in a clay if continued vitrification and shrinkage in

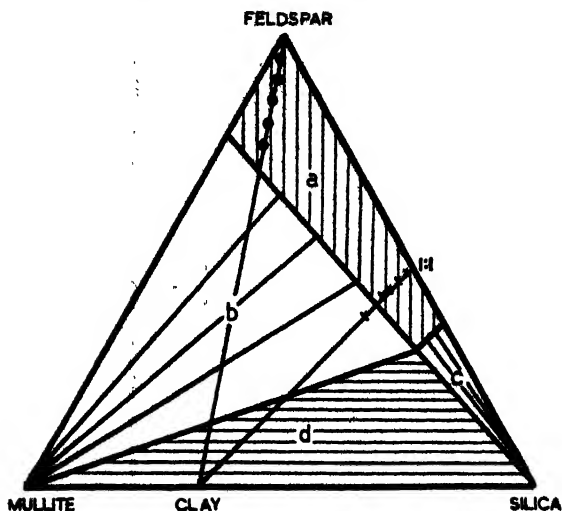


Fig. 125. Effect of batch composition on the constitution of mullite-quartz-feldspar mixtures fired at  $1400^{\circ}\text{C}$ . Area a: glass; b: glass + mullite; c: glass + cristobalite; d: glass + mullite + cristobalite (after H. Navratil and A. H. Fessler).

service is to be avoided. Other procedures tending to give a porcelain-like structure are fine grinding and de-airing.

#### SILLIMANITE, KYANITE AND ANDALUSITE

So great was the shortage of these materials in certain countries during the war, that considerable recourse was had to alternatives, such as china clay calcined at high temperatures. This material consists essentially of mullite and cristobalite and, although less refractory than sillimanite, was used as a satisfactory substitute in a number of applications. The use of aluminosilicate refractories in the 42 to 60 per cent. range is still rather limited in steelplants, but is tending to increase, mainly due to the demand for improved materials for checker top settings, checker chamber roofs and reheating furnace hearths. Bricks for such purposes can be made from any of the above materials, the normal procedure being to grind calcined sillimanite or kyanite to give a 45-10-45 (coarse-medium-fine) type batch, part of the fine fraction consisting of ball clay with sometimes a minor addition of sulphite lye to give green strength. Such bricks are relatively easy to mould, and show a negligible drying and firing shrinkage. After treatment at  $1300^{\circ}\text{C}$ . or higher they develop considerable strength both

in the cold and high temperatures. No difficulty is experienced in getting bricks with a low after-contraction, indeed by a suitable mixture of raw kyanite, bodies showing positive expansion on refiring can be obtained. No matter which of these raw materials is used, the end product after hard firing tends to be mullite and cristobalite, or alternatively mullite and glass. For certain purposes use is now made of raw sillimanite, *e.g.*, the Khasi material, which can be sawn into blocks. No operating data on the use of such materials in steelworks would, however, appear to be available.

Limited use is, however, being made of electro-cast refractories of high alumina content, trials having been made both with the standard mullite composition and with the alternative batches containing zirconia. The procedure adopted for the manufacture of such special materials is well known in connection with work for glass tanks, use being made of three-phase electric arc furnaces. Melting is said to bring about some purification of the batch, *e.g.*, by reduction of iron oxide present to metallic iron. On melting the charge is poured into sand moulds built from slabs of pure sand bonded with linseed oil and supported in a steel frame. The pouring temperature is so high (about 1900°C.) as to make even the tapping of an open-hearth furnace seem a relatively cool operation. Shapes made in this way must be cooled very slowly to avoid strain, larger shapes being covered with insulating materials and left for a week or more before stripping. Bricks of this type consist essentially of mullite, free alumina and a siliceous glass. Their porosity is of the order of 1 or 2 per cent. and they show little sign of softening even at temperatures approaching 1800°C.

#### CORUNDUM

During World War II, rapid developments took place in methods for manufacturing pure fused alumina ware of virtually zero porosity, the principal motive being the desire for super refractories for use in aircraft engines, *e.g.*, and sparking plugs and turbine blades. Although the properties of such refractories are excellent and they are already finding application in steelplants, *e.g.*, in the form of thermocouple sheaths, there seems little likelihood of their general use in brick form in view of the manufacturing techniques involved. These consist essentially in the milling of alumina to an extremely fine condition, followed by slip casting and controlled firing to approximately 1800°C. Where bricks of extremely high alumina content are required, they are generally made by the alternative procedure of bonding fused alumina grain (98 per cent. purity) with a small amount of clay or the like. Bricks of this type fired at high temperatures attain considerable

strength both hot and cold and have properties similar in many ways to those of magnesite bricks. Their price is, however, far greater and their use in steelplants consequently very limited. Considerable use is, however, made of bricks of the 70 per cent. alumina type, made for example by bonding diaspore or fused alumina with larger quantities of clay.

## PROPERTIES OF ALUMINO-SILICATE BRICKS

It is extremely difficult to give a fair picture of the properties of such refractories in view of the great variety at present being manufactured. Tables XXI and XXII will, however, serve to show the kind of product produced in Great Britain and the United States. It will be seen from these tables that porosities varying from 14 to 30 per cent. are usual, and that the refractoriness tends to increase fairly steadily with alumina content. Properties such as cold crushing strength and linear change on refiring are liable to vary considerably even within a consignment, and much more as between the different grades of brick in this particular series. Indeed rather wide fluctuations of such properties as crushing strength and after-contraction are almost a characteristic of fireclay bricks, being far more severe than is experienced, say, with sillimanite or basic refractories.

One of the most notable developments of recent years in this field has been that of the super-duty fireclay brick, which is now produced in large quantities and widely applied in iron and steel works in the United States, and to a less extent elsewhere. Such bricks are fired at a particularly high temperature and are capable of withstanding reheating to high temperatures, provided the temperature cycle itself is not too severe.

Further discussion of the properties of this group of materials will be left to the second half of this book, since such properties can only be discussed in relation to particular applications.

## THERMAL EXPANSION AND PERMANENT LINEAR CHANGE ON REHEATING

Heindl determined the thermal expansion of a wide variety of fireclay bricks up to high temperatures both after medium and high temperature refiring. His curves appear to confirm the general assumption that a firebrick expands just over  $\frac{1}{2}$  per cent. on heating to 1000°C. Above a certain temperature—the precise figure depending on the previous firing treatment—shrinkage sets in and tends to compensate

TABLE XXI  
PROPERTIES OF SELECTED BRITISH ALUMINO-SILICATE BRICKS

Type Code	Siliceous F.45	Medium Alumina F.41	Kaolin F.16	42% Alumina F.22	Sillimanite (62% $Al_2O_3$ ) F.14	70% Alumina X.15	95% Alumina F.7
Apparent porosity, per cent .. .. .	30.6	19.4	24.0	16.4	23.8	24.9	26.2
Bulk density, g.p.ml. . .	1.85	2.08	2.00	2.08	2.33	2.62	2.90
Apparent solid density g.p.ml. . . . .	2.67	2.59	2.63	2.48	3.06	3.48	3.93
Cold crushing strength—lb. per sq. in. Full brick on end . . . .	1940	2500	4890	6050	5250	over 8300	7380
Permeability—perp. 9 in. x 3 in. face—1 skin—c.g.s. units . .	0.011	0.028	0.063	0.071	0.005	0.023	0.011
Permanent linear change on reheating, per cent.	+ 6.5 (exp)	0.0	0.0	+ 0.3 (exp)	—	—	—
2 hours @ 1410°C. . .	—	—	—	—	+ 0.1 (exp)	—0.2 (sh)	—0.3 (sh)
2 hours @ 1500°C. . .	1580	1700	—	1750	—	—	—
Refractoriness, °C. . .							
Refractoriness-under-load—50 lb. per sq. in. rising temp. test:							
Initial deformation . .	1150	1500	1340	1300	1500	1560	1340
Rapid deformation . .	1270	1550	1570	1540	1580	1580	1560
Shear or 10 per cent. collapse . . . . .	1410	1600	1630	1610	1760	1690	1700
Thermal shock resistance	4	30+	30+	30+	30+	30+	30+

TABLE XXII  
PROPERTIES OF SELECTED AMERICAN ALUMINO-SILICATE REFRACTORIES  
(after F. A. Harvey)

	Inter- mediate heat duty	High heat duty	Super duty regular	Kaolin	50% Alumina	60% Alumina	70% Alumina	80% Alumina
Silica content, per cent.	60	53	52	53.5	46	36	22	—
Alumina content, per cent.	35	42	43	43	50	58	72	—
P.C. equivalent	30-31	32½	33-34	33-34	35	36-37	38	—
Approx. °C.	1665	1723	1752	1752	1785	1815	1835	—
Density (bulk), g.p.c.c.	2.114	2.082	2.323	2.082	2.339	2.178	2.082	2.659
Porosity, per cent.	18	18	14	21	16.5	25	35	23
Per cent. spalling loss:								
High duty schedule	—	7.5	0	—	—	—	—	—
Low duty schedule	—	—	2.7	2	2	1	—	0
Reheat test—change-linear:								
5 hours at 1400°C.	—0.5	±0.5	—	—	—	—	—	—
5 hours at 1600°C.	—	—	—0.2	—0.7	+0.5	+0.1	±0.6	0
5 hours at 1650°C.	—	—	—	—2.7	—	—0.4	—	+1.0
Load test—25 lb. per sq. in., 1½ hours, per cent. subsidence:								
At 1300°C.	3.0	—	—	—	—	—	—	—
At 1350°C.	—	5.0	3.0	—	—	—	—	—
At 1450°C.	—	—	5.0	1.0	4.0	—	—	—

for any thermal expansion which may still be taking place. Fig. 126, which shows a continuous expansion-contraction curve for a medium alumina fireclay brick, illustrates the difficulty of discussing the volume stability of fireclay bricks. The particular sample happened to be cut from a fireclay Bessemer tuyere, but could equally well have been part of some other fireclay product. It will be noted that with this sample, the brick showed signs of shrinkage at temperatures as low as 800°C. and that by 1200°C. it had already shrunk 1 per cent.

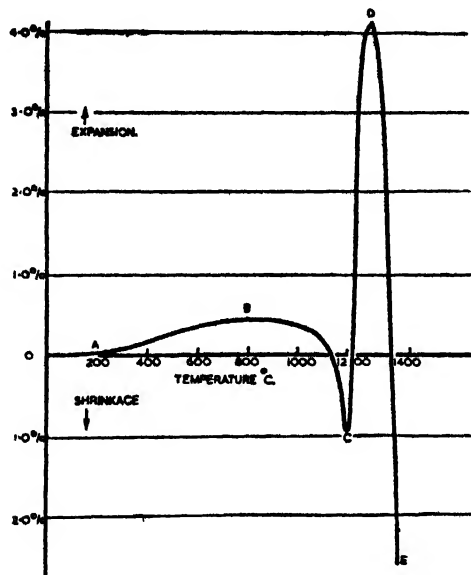


Fig. 126. Continuous expansion-contraction measurements on medium alumina fireclay brick; showing bloating (C-D) after initial thermal expansion (A-B) and shrinkage (B-C); followed by excessive shrinkage (D-E).

Shortly afterwards it started to expand (or "bloat") at a high speed, due presumably to the sealing of the surface and the expansion of trapped gases. On reaching just over 4 per cent., shrinkage again occurred, presumably due to the escape of the trapped gases through the softened envelope. If instead of a continuous curve only "before and after" measurements were available, it is clear that the deduction would have depended greatly on the particular firing schedule. Thus if the test were stopped at 800°C. the brick would probably appear volume stable. Stopped at 1200°C. it would have shown a 1 per cent. shrinkage, whilst a test at 1300°C. would have shown a bloat of over



4 per cent. After-contractions of more than 1 per cent. are likely to lead to trouble in service, due to opening of joints, and consequently an enhanced spalling risk. Bloating on the other hand is not generally serious, particularly since the bricks are normally constrained and any slight bloat that does occur is only in a direction perpendicular to the working face. For certain purposes, *e.g.*, ladles, bloating properties are generally considered advantageous, since they tend to give both tight joints and a dense porcelain-like structure at the working face. A classic example of this type of brick is a ladle brick widely employed in the United States, whose shrinkage on refiring is truly phenomenal (*see* Chapter XIV).

No discussion of the behaviour of fireclay bricks on reheating would be complete without a reference to the work of Clements, whose numerous papers on this subject illustrate the extreme difficulty of getting consistent results for after-contraction tests, even on testpieces cut from the same brick. Just how much of this inconsistency is due to actual variations within the product, and how much due to the extreme difficulty of providing even conditions, both as regards heat treatment and gas atmosphere within a test kiln, still remains to be demonstrated.

#### TORSION TESTS

A vast amount of work has been done by A. L. Roberts and co-workers on the modulus of elasticity in torsion of alumino-silicate refractories, both cold and hot. In 1952 this work on elastic and viscous properties was summarised by Wiechula and Roberts. In this particular paper the authors describe tests carried out on a range of bricks having alumina content from 30.7 to 79.5 per cent. The type of results obtained, which are not unlike those previously discussed for chrome-magnesite bricks, are shown in fig. 127, the particular example being that of the 30 per cent. alumina brick, which incidentally had a cone refractoriness of 30–31 (1670° to 1690°C.) and showed 10 per cent. deformation in the refractoriness under load test at 1575°C. under a load of 50 lb. per sq. in. Its cold crushing strength is 5600 lb. and its thermal shock resistance very good. It will be seen that up to about 600°C. it behaves in a reasonably elastic manner, returning to its original position when the load is removed, but that above these temperatures there is a marked hysteresis effect, which becomes more substantial at temperatures of 1000°C. and over. Information of this type is extremely valuable, in that it shows that that part of a structure built of such bricks, which is above 800°C., is incapable of carrying a load for any period. Even if initial arch stresses for example are concentrated at the hot face, creep at this point will soon result in the load

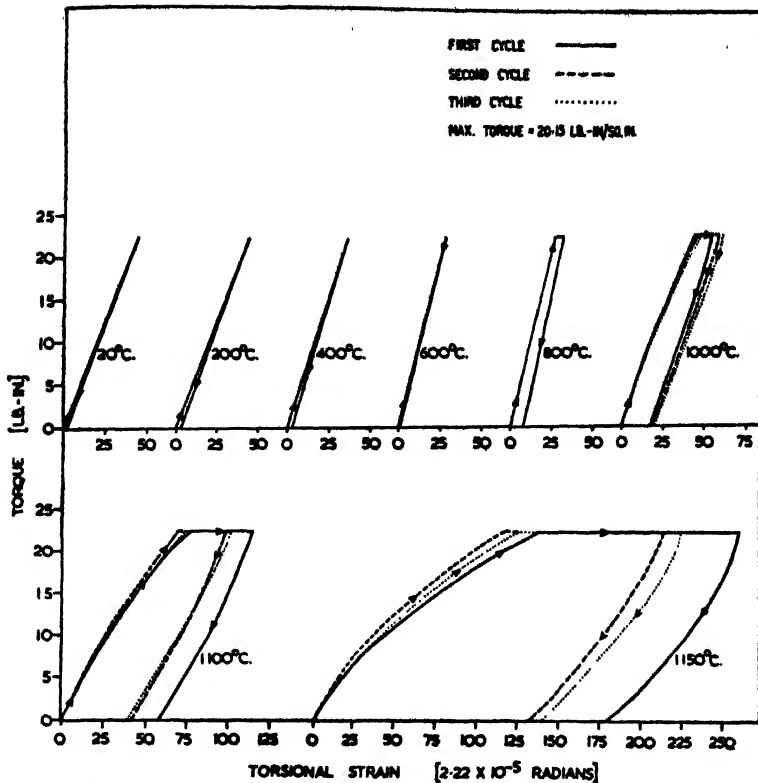


Fig. 127. Stress-strain curves obtained in torsional modulus tests for 31% alumina fireclay brick (after Wiechula and Roberts).

being transferred to the cooler part of the brick. In the same paper the authors give the results of ultimate shear strength (see fig. 128) as a function of temperature for a range of aluminosilicate bricks, starting with silica and going to one containing nearly 80 per cent. alumina. The curves show something which has long been recognised, though never before demonstrated in such detail, *viz.*, that at temperatures around 800°C. fireclay bricks are actually stronger than they are when cold, but that above this temperature their strength falls off very rapidly. Given such information it is not difficult to appreciate the risk of over-insulating an arch built of fireclay bricks. The fact that such bricks showed marked creep above 1000°C. also presumably means that the sudden cooling of complete bricks from high temperatures down to this level is less likely to result in undue thermal shock.

In other papers Roberts and his colleagues have shown the effect of pressing, *e.g.*, plastic and dry press methods, on the rigidity and hence

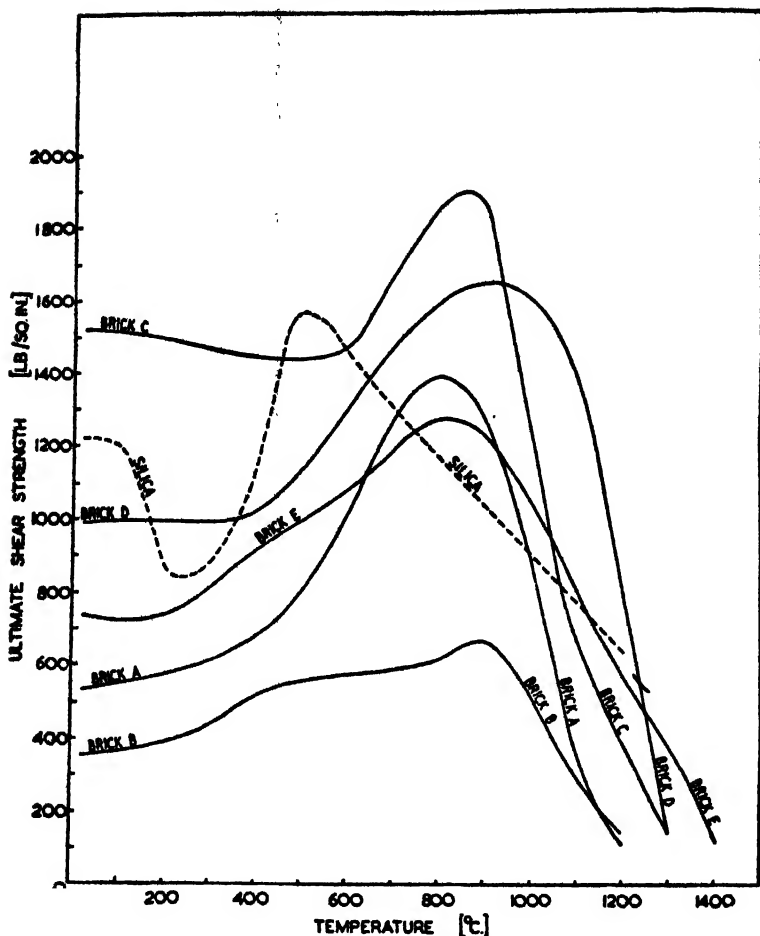


Fig. 128. Ultimate shear strength curves for alumino-silicate refractories. Alumina contents as follows: brick A: 31%; B: 43%; C: 58%; D: 73%; E: 80% (after Wiechula and Roberts).

on the thermal shock resistance of various alumino-silicate refractories. Those who wish to follow the matter further, are recommended to study the original papers, some of which are cited in the bibliography.

#### SPALLING RESISTANCE

The factors controlling spalling of any refractory brick are numerous, and those of alumino-silicate refractories particularly difficult to understand, in view of the low temperature at which softening occurs.

The knowledge that spalling behaviour is greatly influenced by previous treatment has led to such treatment being included in the A.S.T.M. Panel Spalling Test (C38-49), which comes nearer to simulating service conditions than any other spalling test at present in use. That such changes are important has been observed by many workers in iron and steelplants, where surface vitrification of the working face has frequently been observed to lead to separation from the cooler portion of the brick.

What is still badly required is the spalling test which would enable fireclay bricks to be tested under simulative service conditions without the recourse to such a long and expensive procedure, together with an explanation of the observed behaviour of such tests in terms of fundamental properties, such as specific thermal shock resistance and after-contraction.

#### SLAG RESISTANCE

The resistance of aluminosilicate refractories to slags high in iron and manganese has already been discussed in connection with the corresponding oxide systems. In normal laboratory work, however, more direct tests, such as the determination of the melting point of brick-slag mixtures, the action of a pellet of slag when melted on a fireclay brick, or the effect of a gas flame loaded with slag particles when allowed to impinge on the brick surface, are usually employed. Attempts have been made, *e.g.*, by Fehling, to produce the problem more fundamentally, and to define the attack in terms of viscosity of the slag and the solubility of the refractory in the slag at the working temperature.

The latter is important, not because the slag ever succeeds in dissolving enough brick to reach equilibrium, but because it is the solubility of the brick in the slag which determines the gradient of concentration in the reacting layer, and hence the rates of diffusion of the reaction products. Fehling's studies were made on the attack of coal ash on fireclay bricks, but similar considerations would apply with other slags and refractories. One of the principal difficulties in all such studies, is that steelworks slags normally contain considerable amounts of iron-oxide, whose corroding influence is greatly influenced by its state of oxidation. Furthermore the precise atmospheric conditions in furnaces even when known are usually very variable and, therefore, difficult to simulate.

Slag tests on aluminosilicates must, therefore, be considered as mainly of value, in enabling technologists to sort out those bricks

which warrant trial on a larger scale. Meantime the collection by numerous firms of both laboratory and performance data on such bricks, should enable the prediction of behaviour in the plant from laboratory tests to be made with increasing certainty.

## CASTABLES, PLASTICS AND CEMENTS

During recent years there has been a marked expansion in the use of plastic ramming mixtures and castables made from crushed firebrick, with added bond, such as plastic clay or aluminous cement. Certain of these materials have been found to be so durable that they are even specified for such vulnerable positions as fantail arches joining the slag pockets to the checker chambers of open-hearth furnaces. Most of the plastic material is supplied in water-proof containers, in a ready to be used condition, whilst the castables normally merely require the addition of water to develop the hydraulic properties of the added cement. For many purposes the presence of large amounts of lime, and the consequent lowering in melting point of alumino-silicates, would prevent their use, but for others, *e.g.*, gas ports on soaking pits, where the temperatures are not particularly high, and large shapes are desirable, the use of such hydraulic setting material can be highly advantageous.

These developments are not new, a fairly full description being given for example by Hussey in 1937 of the ciment fondu bonded type. The precise facts are still somewhat shrouded by the proprietary nature of most materials in this category, but reference to the articles quoted in the bibliography provides a fair impression of the mixes used and the properties obtained.

Most fireclay bricks are set with a cement consisting either of fireclay, or of fireclay with an addition of some finely ground grog, which may be firebrick itself or possibly sand. If the bricks are of excellent shape they may be set dry, or merely dipped in finely ground clay slip. For other purposes, buttered joints are preferred. The disadvantage of straight fireclay cements is their high shrinkage on drying and firing, and relatively low strength in the unfired condition. For this reason special cements, *e.g.*, those made from calcined diaspore, clay and water glass, are frequently preferred, in spite of their much higher price. Indeed it is true to say that the ease with which such material can be used, and the excellent results obtained, often leads to the use of these special cements in positions where they are neither necessary, nor economic. Some of these cements in spite of their sodium silicate content, have a refractoriness of 1690°C. or over, and, due to the presence of a

considerable proportion of precalcined material, are relatively free from drying and firing shrinkage.

What must be stressed is that the mere fact that the cement is easy to work, has a long water retention time, and does not crack on drying, is no justification for its use with refractories for which it was never intended. Alumino-silicate cements are principally designed for use with alumino-silicate refractories, and can do considerable damage if used, for example, with silica bricks.

## APPLICATIONS


The principal applications of alumino-silicate refractories in steelplants are discussed in some detail in the chapters dealing with furnaces and the casting pit. It is worth noting, however, that they find many other uses in the iron and steel industry, particularly for the construction of blast furnaces and the cooler parts of coke ovens. They are also used in substantial quantities in cupolas, which although frequently found in steelplants, are more strictly part of the technology of iron making. Here they are frequently protected by a covering of ganister (quartzite-clay) which has to be renewed at frequent intervals.

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## Chapter VII

# INSULATION

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LIVER LYLE IN DISCUSSING LAGGING in his book on *The Efficient Use of Steam*, quotes a delightful parody on Hiawatha:

“With the skin he made him mittens,  
Made them with the fur side inside,  
Made them with the skin side outside.  
He, to get the warm side inside,  
Put the inside skin side outside;  
He, to get the cold side outside  
Put the warm side fur side inside.”

This appreciation of insulation should at least serve to introduce the serious and oft neglected question of heat conservation. At a time when we in Great Britain are at a loss to know how to mine enough coal, or alternatively find enough foreign exchange to buy oil, we are still allowing a lot more heat to leak through furnace walls than is strictly necessary.

Insulation does not make any vast difference to the overall efficiency of either the open-hearth or the reheating furnace, but it does enable the losses through certain parts of the structure to be halved and thus save amounts of heat that frequently more than pay for the extra building costs involved. Insulation rarely adds to refractory life, and may even reduce it. It is not for this reason particularly attractive to the refractories engineer. If, however, as is increasingly the case, he is willing to use corrected refractories costs that take account not only of bricks and labour for building, but also of credits or debits due to changes in output and fuel consumption, then a case for insulation can frequently be made. With reheating furnaces, where hot face insulation—“with the fur side inside”—is practicable, substantial savings can also accrue due to reduced heat storage.

Whether insulation is justified or not depends on:

- (a) The anticipated fuel saving—which can sometimes be calculated and occasionally checked.
- (b) The increase (or decrease) in the output of the furnace.

- (c) The increase (or decrease) in refractories costs—including insulation.
- (d) The cost of alterations to foundations or steelwork necessary to permit insulation.

The insulation of reheating furnaces is almost always justified, and any plans for new furnaces, which do not include insulation should be viewed with suspicion, since although they may mean a lower quotation, they almost always mean a greater overall operating cost. This fact has been demonstrated so many times both in theory and practice that it scarcely warrants further discussion. What does require further consideration is whether certain reheating furnaces, now externally insulated, should be built with hot face construction.

The insulation of open-hearth furnaces, or other steelmaking units operating at very high temperatures, is always a compromise. There is no doubt that an initial heat saving results, but where refractories, in particular silica bricks in roofs, are already operating close to their maximum safe temperature, the amount of damage done by a short-term overheat is much greater with an insulated roof than with an uninsulated one. It should also be noted that whereas the measuring point may be operating at a given temperature, other regions on the same surface may be hotter or colder, and the difference vary substantially even within a heat. As a general guide parts which are already subject to heavy wear should be left uninsulated, the initial application being applied to parts whose thickness is relatively constant throughout the campaign.

## RAW MATERIALS

As Fehling has pointed out, all the known insulating materials derive their low thermal conductivity from the air enclosed in their pores, whilst their heat capacity is determined almost entirely by the solid component. The insulating effect is principally the result of achieving a series of air spaces between an alternate series of solid boundaries. Clearly the more air present, and the less solid, the lower will be the conductivity, and hence such correlations as were noted in Chapter I between porosity and conductivity are to be expected.

## DIATOMITE

One of the most commonly used insulating materials, *viz.*, diatomite, or "kieselguhr" as it is sometimes called, achieves the above condition by making use of a mass of minute aquatic plant skeletons deposited thousands of years ago on the beds of seas and lakes. Some 8000 different



species have already been identified, their shapes and sizes being so characteristic that experts can frequently tell the source of a material merely by microscopic examination. Fig. 129, p. 283, illustrates the structure of a particular diatom—highly magnified—the original diatom having passed through a 250 mesh screen. It will be seen that there are numerous gaps within the skeleton, in addition to those formed between the individual skeletons themselves. Although silica is the principal constituent in diatomite, it is by no means the only one, considerable amounts of combined water, organic matter, and other impurities, notably clay, being present. Table XXIII gives chemical analyses of materials from different parts of the world, from which it will be seen that some are much purer than others. The presence of impurities of the clay type, together with the extremely high initial porosity, makes such materials very susceptible to shrinkage on heating and they are, therefore, rarely recommended for use at temperatures approaching their final melting point, which generally lies in the range  $1400^{\circ}$ – $1700^{\circ}\text{C}$ . Incidentally, diatomite is usually regarded as amorphous, but X-ray examination shows that this is not always the case, beta cristobalite having, for example, been detected.

Among the larger deposits are those in California, where white coloured sawn blocks are produced, Denmark (the principal British source), Germany, the U.S.S.R., Portugal and Northern Ireland.

#### CLAY

High temperature insulating bricks are made both from china clay and from less refractory fireclays. These materials have already been described in Chapter VI on aluminosilicates.

#### SILICA

Here again the material is similar to that used for the production of refractory bricks (*see* Chapter II), the main difference being the much finer grinding and the subsequent processing.

#### ASBESTOS

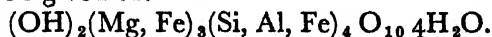
Marco Polo, the explorer, can claim to be among the pioneers in the insulation field, since it was he who discovered asbestos being used, as fire-resisting cloth, in Siberia, as far back as the 13th century. Mineralogically, asbestos belongs to one of two main groups, the amphiboles and the hydrated magnesium silicates of the serpentine type. These minerals, although mainly magnesium silicates, often contain considerable amounts of lime and iron oxide. The asbestos used industrially,

chrysotile, which belongs to the serpentine group, constitutes by far the largest part. This material is found in considerable quantity in Quebec, in the State of Georgia (U.S.A.), in Southern Rhodesia and in the U.S.S.R.

The fibrous nature of asbestos and its ability to crush without crumbling, are marked advantages with certain types of installation, *e.g.*, steam pipe lagging and the insulation on blast-furnace stoves. On the other hand its crushing strength in brick form is usually very low.

#### VERMICULITE

The mineralogy and fundamental structure of this micaceous type material, has been fully reviewed by Cowling and White, and those who are interested in this aspect are advised to read the original paper. The term mica is, of course, applied to a whole range of silicates of alumina with potash and hydroxyl. If the potash is dominant, the material is known as a potash mica (muscovite), whereas if lithia is dominant it is known as lithia-mica (lepidolite). Vermiculite, also called jefferisite, is a hydrated biotite (magnesia-iron mica) whose theoretical formula has been given as:



In its natural form vermiculite is a flaky and quite solid material, but on heating suddenly it exfoliates, *i.e.*, the solid flake bursts into a large number of individual flakes separated by air spaces. The final appearance of a well exfoliated grain being as shown in fig. 130, p. 284. Examination of this is sufficient explanation of the name, which derives from the Latin *vermicularis*, meaning wormlike. If only single grains are considered, such a material would clearly approximate to the ideal set by Fehling of a large number of air layers in series.

Numerous methods have been suggested for the exfoliation process, but the one generally adopted is sudden heating in some type of furnace, *e.g.*, rotary, cascade, vibrating tray or injection type. Satisfactory exfoliation depends, as Cowling and White emphasise, on the application of the heat in such a way that the water between the plates is rapidly converted into steam and has, therefore, the maximum disruptive effect on the structure. The initial dehydration has been shown by these authors to be reversible, but if the material is heated strongly enough to cause even partial irreversible dehydration it becomes weak and friable. The time taken in exfoliating should therefore be as short as possible, and given adequate control, temperatures from 750° to 1300°C. can be used. The granules so formed are the raw material for the subsequent production of loose fill (cements) or bricks.

One of the first deposits to be exploited commercially was that at Libby, Montana, but most of the material now used in Great Britain comes from South Africa. Chemical analyses of vermiculite and hydrobiotite samples from Baltimore and from Montana (U.S.A.), and from Palabora (South Africa) are seen to cover the following composition range:

MgO	..	..	..	..	24-28
Al <sub>2</sub> O <sub>3</sub>	..	..	..	..	11-19
SiO <sub>2</sub>	..	..	..	..	36-44
Fe <sub>2</sub> O <sub>3</sub>	..	..	..	..	4- 8
Alkalis	..	..	..	..	up to 5%

In addition there is a water content of 10-19 per cent.

#### SPECIAL ALUMINO-SILICATES

Special type insulating bricks are made from sillimanite and kindred materials, the properties of which have been discussed in Chapter VI. One of the latest developments in this field is the use of alumina bubbles made by a patented process.

#### BASIC

A number of basic materials, in particular magnesite and chrome-magnesite, have been made up in insulating form. Bricks have also been made from raw dolomite mixed with silica, and from serpentine-magnesite mixtures by foaming or the addition of combustibles. The raw materials in all cases are those used for the production of the corresponding basic refractories. One of the latest additions to this group consists of stabilised zirconia, produced in porous condition, and consequently capable of being made into relatively light-weight bricks. Experimental batches have also been made of the spinel type, by using fired alumina grain with raw magnesite.

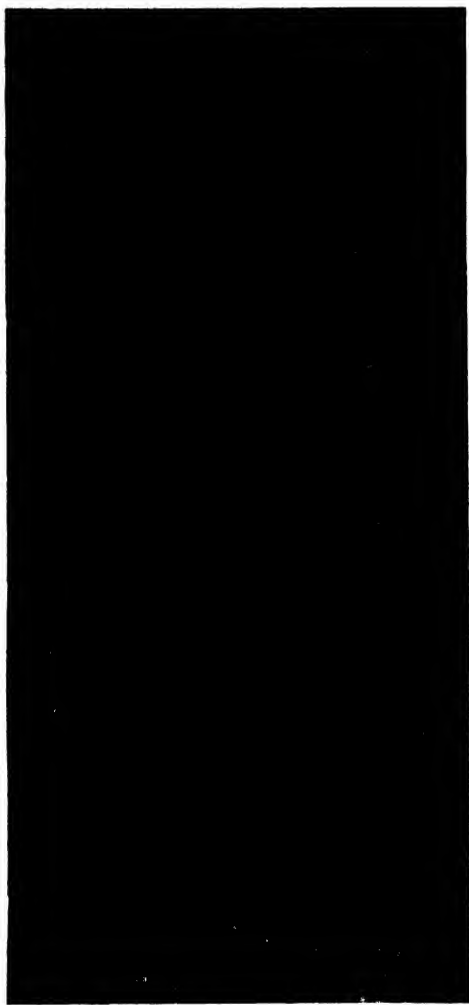
#### BRICK MANUFACTURE

The methods employed in producing insulating bricks are so extensive that they could in themselves constitute a small book. In the present section, therefore, only a summary of the position can be made.

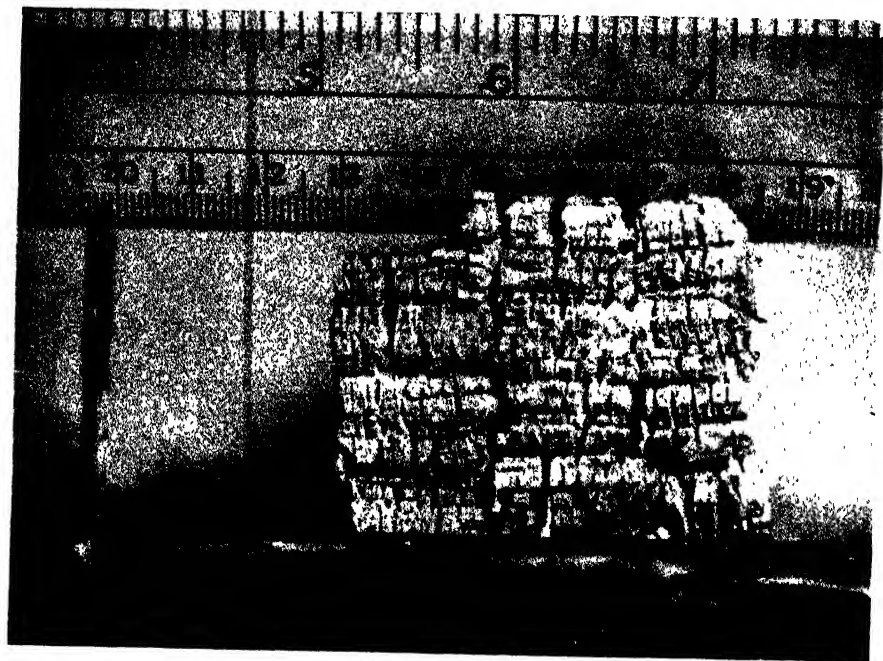
#### DIATOMITE

Oliver has summarised the manufacturing methods used as follows:

- (1) Sawing into blocks directly from the face of the deposit—to give a “natural” grade.



*Fig. 129. Diatom showing minute cellular structure (after Oliver).*



*Fig. 130. Large flake of raw vermiculite (left) and similar but exfoliated grain (right) (after Cowling and White).*

- (2) Natural grade blocks heated to about 950°C. to make them suitable for use up to 800°C.
- (3) Moulding, light pressure or extrusion, of wet diatomaceous earth with or without clay additions followed by firing to suitable temperature.
- (4) The addition of combustibles, *e.g.*, ground cork, charcoal or sawdust, to a diatomaceous earth batch.
- (5) The bonding of precalcined diatomite granules with plastic clay followed by firing to a relatively high temperature.

Although an extraordinary variety of diatomite bricks are produced by the above processes, they can still be roughly divided into two types:

- (1) Bricks having high insulating quality but a relatively low mechanical strength.
- (2) Bricks in which low conductivity is sacrificed to improve mechanical strength.

This latter group is sometimes referred to as "solid grade".

Diatomite presents a rather unique problem, both as regards drying and firing, in that the batch may contain as much as 50 per cent. of moisture, which must be removed in tunnel or chamber drying before setting in the kiln, and that such porosity inducing media as sawdust and ground cork, being themselves fuel, make very difficult the maintenance of a strict firing schedule. In addition the raw diatomite itself often contains substantial quantities of carbonaceous material.

Because of the high total shrinkage from moulding to fired, bricks are usually made in relatively rough clot form and are machined to agreed size and shape after drying and firing. Special shapes are normally made by cutting from large lumps of fired material.

#### CLAY

The production of insulated bricks from materials of a fireclay type, is carried out by a number of different methods. These have been summarised by Clements, Barrett and Green as follows:

- (1) The incorporation of natural or synthetic light-weight materials, such as diatomite, expanded vermiculite or raw kyanite.
- (2) The inclusion of materials which are later removed by combustion or sublimation—the former being more usual.
- (3) The production of a cellular structure by mechanical beating using a frothing agent.
- (4) The formation of gas bubbles in a semi-fluid medium by chemical reaction.
- (5) Miscellaneous methods.

Although certain of the above processes would seem comparatively simple, the bulk production of porous clay insulating bricks of low conductivity is, in fact, a very tricky operation, that is frequently associated with considerable losses both of bricks and money. Of the above methods the use of combustibles is probably the earliest and the most widely employed. The particular material used is partly a function of the water required in the product and partly of availability. Sawdust is perhaps the most common material, though better results are said to be obtained with brazil-nut shells. Even such simple things as sawdust require careful selection and control, since such factors as the grain size and swelling tendency in contact with water may have a marked effect both on the ease of manufacture and the properties of the product. Given the right batch the brickmaking may proceed by slop or hand moulding, by extrusion or by semi-dry or dry pressing. Theoretically the formation of a bubble structure has much to commend it, in that no ash remains in the product to reduce its refractoriness, whilst adequate control of bubble size, together with the resulting close pore structure, should provide an insulator close to the theoretical ideal.

The drying problem is similar to that of diatomite, but the firing problem more difficult, in that higher temperatures are normally desirable for this type of product. The tendency to deform under load at high temperatures of such comparatively weak bricks does, however, severely limit the height that can be used in the kiln setting—a problem sometimes answered by the use of tunnel kilns.

#### SILICA

As far back as 1896 a method was patented for producing a porous silica brick by the admixture of paper-making products with ground ganister or other siliceous material. Very little appears to have been published from this time on, though a number of firms did produce porous silica bricks made either by the addition of combustibles or by a special frothing process. One such was, for example, used in Sweden, where an addition of lime and aluminium were made to the silica batch, the hydrogen generated by the reaction being sufficient to raise the material as yeast raises dough. Given this expanded product, bricks made from it are dried and fired in the same manner as with ordinary silica bricks. Where combustibles are used, the porosity is normally only of the order of 50 per cent., compared with say 70 per cent. for bricks made by the foaming method.

### ASBESTOS

Although relatively little appears to have been published on the methods used in manufacturing such bricks, considerable quantities are in fact sold, either in brick or special shape form, and with varying amounts of fillers, such as calcined diatomite mixed with asbestos fibre.

### VERMICULITE

Vermiculite is usually employed, mainly in the form of loose granules, for packing gaps between say brick linings and a steelcase, or as a material that can be plastered or spread on to the outside of a structure. A limited quantity is, however, made into bricks by the addition of small amounts of bond (presumably of the clay type), light moulding and firing to develop strength. As with so many other insulating bricks, the final shape is got by machining, whilst many sizes are prepared by sawing a number of units from a relatively large block of fired material.

### SPECIAL ALUMINO-SILICATES

It has been demonstrated both by research workers and by production plants, that very excellent insulating bricks can be manufactured from a sillimanite type base. Thus Salmang and Frank reported the production of bricks varying in porosity from 45 to 63 per cent., whilst Clews, Dobbins and Green, reported results on sillimanite bricks made by adding anthracite, or paper pulp, as combustibles, or using aluminium and acid to get froth. As with silica bricks, a high firing temperature—of the order of  $1400^{\circ}\text{C}$ .—is required in order to get the best properties in a product.

### BASIC

Here again numerous research workers have shown the possibility of making bricks, particularly dolomite and chrome-magnesite, but the quantities of material produced commercially are negligible. One brick made by a German company from a hard-fired Grecian magnesite, and having an intermediate porosity, was demonstrated as having a high durability even as a hot face material in an open-hearth furnace. With materials containing chrome, the complications arising when reducing conditions exist have led to attempts to make bricks by foaming: thus Chesters, Howie and Lynam, were able to mix foams made by whipping a detergent solution with finely ground chrome-magnesite slurries and obtained satisfactory bricks, provided the latter were allowed to stand for a reasonable period in the mould before stripping. Faster removal can be achieved by the addition of setting



agents, but the effect of these on the refractoriness of the ultimate product cannot be ignored. Using such procedures bricks of porosities as high as 60 per cent. were obtained.

An alternative method, which is applicable to both chrome-magnesite and to magnesite-serpentine batches, is the use of a raw magnesite instead of dead-burned, which automatically yields porosity but also physical weakness due to loss of carbon-dioxide. The same principle was used by Chadeyron and Rees in an extensive investigation on porous dolomite bricks. These authors employed a mixture of raw dolomite with 14 per cent. of added silica to convert the lime to dicalcium silicate, which was then stabilised by the use of suitable additions.

With all such bricks, the possibility arises of making a product with a relatively hard and solid face and a relatively porous but weak back. Thus bricks having a face similar to a firebrick and a back similar to a high temperature porous fireclay brick, have been produced for use in reheating furnaces, where abrasion or dust pick-up make the use of straight hot face insulation impracticable. Preliminary experiments suggest, however, that such bricks are liable to loss of the working face ( $\frac{1}{2}$  in. to 1 in.), presumably due to a stress concentration at the interface following frequent heating and cooling. For most purposes the more practical procedure would appear to be the use of a hot face insulating brick with a single or multiple coating of hard refractory cement.

## PROPERTIES

Insulating bricks are often placed in simple categories, *e.g.*, low, medium or high temperature classes, although such divisions are arbitrary they can prove convenient. In an earlier classification a range of 900° to 1200°C. was employed for the medium temperature material; low temperature insulation and high temperature insulation falling below and above this range. Quite recently a new system, *viz.*, the A.S.T.M. classification, is becoming popular. This suggests the following temperature ranges:

Group identification	<i>Reheat change not more than 2 per cent. when tested at</i>	<i>Bulk density not greater than</i>
16	1550°F. ( 845°C.)	34 lb. per cu. ft.
20	1950°F. (1065°C.)	40 " " " "
23	2250°F. (1230°C.)	48 " " " "
26	2550°F. (1400°C.)	52 " " " "
28	2750°F. (1510°C.)	60 " " " "

The "Group Identification" number multiplied by 100 represents the maximum temperature to which the hot face of the brick may be exposed in service. Thus the bricks in group 16 may be heated to 1600°F., *i.e.*, belong to the low temperature class, as defined above. Even this range is incomplete since there are commercially available insulating firebricks capable of working up to 3000°F. (1650°C.) and light-weight silica bricks specified for use to 2900°F. (1595°C.).

Bricks of the diatomite, asbestos and vermiculite type almost all fall in the low temperature category, though a few specially prepared diatomite bricks can be used at temperatures up to at least 1100°C. Bricks in the high temperature class consist in the main of porous fire-clay, together with smaller quantities made from silica, sillimanite and basic materials.

#### TEST METHODS

The methods used in testing insulating bricks are very similar to those described in Chapter I in connection with refractories. The tests for porosity, permeability and cold crushing strength, for example, are identical, though alternative bulk density methods to water soaking, *e.g.*, mensuration, may be necessary with asbestos type materials, due to the risk of distortion. Where refractoriness under load tests are employed, the dead load is normally reduced to 10 lb. per sq. in. maximum, instead of the usual 25 or 50 lb. per sq. in. This takes

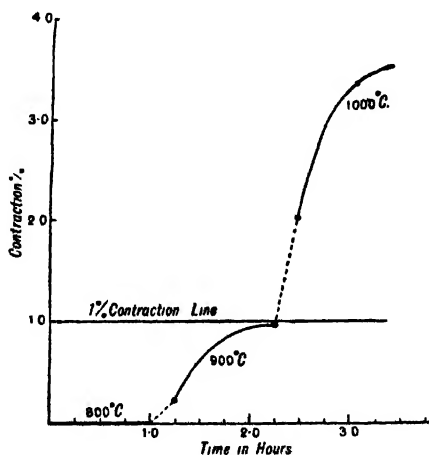


Fig. 131. Continuous after-contraction curve for diatomite brick, showing the effect of approximately 1 hour at 800°, 900° and 1000°C.

account of the lower inherent weight of walls constructed from such materials. The small prism test for thermal shock is only of value in showing the extreme difference, *e.g.*, between aluminous and siliceous grades of high temperature insulating brick, and no satisfactory means of predicting spalling behaviour in service for insulation has as yet been reported. Thermal conductivity is tested by the methods described in Chapter I, but some useful results on permanent linear change on reheating may be obtained by departing from the standard procedure in favour of a continuous test, in which a sample of the brick is heated up at a steady rate in thermal expansion apparatus and held at various temperature levels, *e.g.*, 1 hour at 800°C., 1 hour at 900°C. (*see* fig. 131), so that its volume stability at these temperatures may be assessed. The temperatures above which rapid shrinkage is likely to occur can thus be determined and failures of the type illustrated in fig. 132, p. 293, eliminated.

#### RESULTS

It is found convenient to report the results of tests on insulating bricks on a standard form of the type shown in fig. 133. It is relatively easy

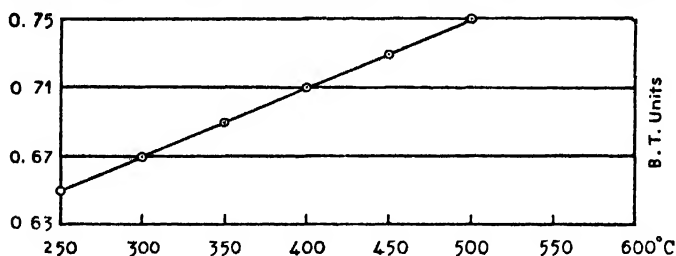


Fig. 133. Test data on Insulating Bricks.

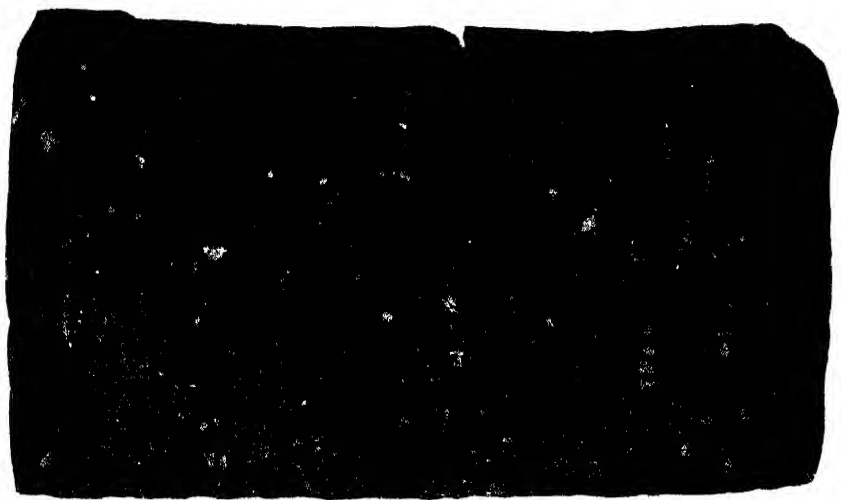
when the duty for which a brick is required has been specified to select from such sheets the few bricks which appear suitable, and then to investigate further their relative merits, *e.g.*, comparative thermal conductivities, cold crushing strength and maximum safe temperature, in relation to the expected duty. Finally other relevant information, *e.g.*, the price of the bricks selected, can then be obtained and a final recommendation made. The risk that deliveries may not match up to test samples is not perhaps as great for insulating bricks as it is for certain other furnace materials, since the deliberate selection of, say, light weight samples by the supplier, although yielding good conductivity figures, is likely to mean low crushing strengths, whilst the selection of a high strength brick is likely to show a high conductivity. It is for

TABLE XXIII  
TYPICAL ANALYSES OF DIATOMACEOUS EARTHS  
FROM IMPERIAL INSTITUTE—"THE MINERAL INDUSTRY OF THE BRITISH EMPIRE AND FOREIGN  
COUNTRIES. DIATOMACEOUS EARTH",

	<i>Northern Ireland</i>	<i>German (Steinbeck)</i>	<i>Denmark</i>		<i>Algeria (St. Denis du Sig)</i>	<i>Spain (Elche de la Sena)</i>	<i>U.S.A. (Lompoc)</i>
			<i>(Mors) Raw</i>	<i>(Fur) Dried</i>			
Silica ( $\text{SiO}_2$ ) ..	73.01	73.16	47.03	75.8	72.9	80.30	88.78
Alumina ( $\text{Al}_2\text{O}_3$ ) ..	8.55	4.86	6.21	9.0	2.4	0.83	2.68
Iron oxide ( $\text{Fe}_2\text{O}_3$ ) ..	2.09	3.04	5.46	4.0	0.9	0.16	Trace
Titanium dioxide ( $\text{TiO}_2$ ) ..	—	—	—	—	0.3	—	—
Lime ( $\text{CaO}$ ) ..	1.14	0.85	0.66	0.9	7.3	3.59	1.61
Magnesia ( $\text{MgO}$ ) ..	0.83	—	0.87	1.3	2.4	0.49	1.30
Alkalis ..	0.28	—	1.21	1.0	—	0.82	—
Organic matter and com- bined water ..	7.71	14.47	8.34	7.0	4.8	7.06	—
Moisture ..	6.39	—	30.25	—	—	6.69	5.54
Sulphuric anhydride ( $\text{SO}_3$ ) ..	—	—	—	—	—	—	—
Carbon dioxide ( $\text{CO}_2$ ) ..	—	—	—	—	7.9	—	—
Undetermined ..	—	—	—	—	1.1	—	—

TABLE XXIV  
PROPERTIES OF DIATOMITE INSULATING BRICKS

Code No.	Thermal conductivity at 400°C. (Mean) (British Thermal Units)	Maximum safe temperature °C.	Cold crushing strength ( $9'' \times 4\frac{1}{2}'' \times 3''$ bricks) on end lb. per sq. in.	Porosity per cent.	Bulk density		Permeability through 1 skin e.g.s. units
					g.p.-ml.	lb. per cu. ft.	
1	1.30	—	130	69	0.81	50.7	0.029
2	3.16	> 1200	380	54	1.24	77.1	0.140
7	0.59	900	240	74	0.52	32.4	0.0006
8	1.74	1000	400	52	1.09	67.7	0.050
9	1.00	950	300	65	0.73	45.3	0.011
10	0.70	950	170	77	0.51	31.6	0.078
19	0.87	875	60	80	0.44	27.2	0.188
20	0.98	800	180	77	0.54	33.5	0.124
21	0.84	850	140	74	0.55	34.1	0.180
24	1.55	900	740	68	0.85	53.1	0.013
25	0.91	875	300	77	0.53	33.1	0.031
26	1.18	1100	980	60	0.64	41.6	0.05
37	1.22	850	410	66	0.74	46.1	0.003
44	1.12	950	490	65	0.76	47.5	0.002
47	2.10	> 1200	200	65	0.92	57.5	0.66
49	1.66	1000	210	65	0.91	56.8	0.267
50	1.23	950	230	68	0.77	48.0	0.025
51	2.00	1100	550	68	0.80	50.0	0.057
52	1.24	900	210	66	0.86	54.2	0.013
56	1.29	900	180	69	0.77	48.0	0.02



*Fig. 132. Diatomite brick shrunk and cracked due to overheating of  
9 × 3 in. face.*



this reason in the suppliers own interests that the bricks chosen should be typical of the delivery, and this can best be assured by choosing those of average weight or bulk density.

### *Diatomite bricks*

Since the data shown in Table XXIV were obtained, an additional 15 diatomite bricks have been tested in the same laboratory. The fact that the properties of these all fall within the range already given in Table XXIV, suggests that there has not been much change in diatomite bricks during recent years, and that the wide variation in properties shown is also likely to be found in practice. A study of this table shows that merely specifying a "diatomite brick" is quite inadequate, since such a brick may have a conductivity from 0.6 to 3.2 B.Th.U. at 400°C. (mean), with a maximum safe temperature anywhere between 800°C. to over 1200°C., and a cold crushing strength varying from 60 to 740 lb. per sq. in.

The first selection is usually made on the basis of maximum safe temperature, which must however be considered in conjunction with conductivity, since diatomite bricks are rarely used in the working face, and the interface temperature between the refractory lining and the insulating backing cannot be estimated until the conductivity is known. Thus it might be shown that brick No. 26, with a conductivity of 1.18 would have a hot face temperature of 950°C. if used in a particular arrangement. Since its maximum safe temperature on test was found to be 150°C. higher than this and its crushing strength very good, the brick should prove quite satisfactory in this position. Its other properties, e.g., bulk density, are then largely of academic interest, since insulating bricks are bought by the thousand and their weight is not therefore of any particular significance. It is, however, interesting to note that a fairly close correlation (coefficient 0.89) exists between the thermal conductivity and bulk density of the diatomite bricks listed in Table XXIV. Fig. 29 shows that an estimate of the conductivity from the bulk density is unlikely to be widely in error, though Cowling, Elliot and Hale, have pointed out that the relationship with refractory insulating bricks is nowhere near as close, and that for example the conductivity of a brick of 30 lb. per cu. ft. density might be anywhere between 1.20 and 1.62 even though it came from the same consignment. They found, however, that the error was greatly reduced if the average bulk density of nine samples were taken rather than a single brick.

The answer to the question of whether diatomite bricks lose their insulating value in service, has been given by Oliver and Rigby, who



described the effect on their properties, in particular conductivity, of refiring for 6 hours at 950°, 1000° and 1050°C. Although the results (as set out below) refer to one particular brand of brick, they should at least serve to show the dangers associated with overheating. In practice, of course, such overheating is normally limited to the working face and the drop in effective conductivity is, therefore, very much less than that shown:

<i>Refiring temperature</i>	<i>Percentage shrinkage (average—linear)</i>	<i>Percentage increase in bulk density</i>	<i>Percentage increase in thermal conductivity at 450°C. mean</i>
950°C.	2.03	6.2	6.95
1000°C.	2.44	9.5	16.90
1050°C.	4.27	14.8	30.60

#### *Fireclay insulating bricks*

With diatomite nature has provided the small pores, but with fireclay insulating bricks man must find means of raising them from the 25 per cent. that is all too easy to get, to the 60 to 70 per cent. that tends to make a very weak brick unless special techniques are employed. In general the additional pores, which should preferably be small and sealed, are obtained by adding finely ground combustibles, such as anthracite to the batch, or alternatively, and much less usual, by an aeration process. Table XXV gives the properties of a number of clay base British insulating bricks, from which it will be seen that maximum safe temperatures as high as 1500°C. can be obtained with bricks having porosities as high as 68 per cent. Furthermore that such bricks may have cold crushing strengths even in the on-end position of over 350 lb. per sq. in. The production of such a brick is a triumph of the ceramic art, necessitating not only a first-class raw material but first-class techniques in manufacture. It is not surprising that such bricks, although feather-light, are far more expensive than their solid counterparts. The typical conductivities for such materials are roughly double those of the diatomite type, but this is offset by the great value of being able to employ them as hot face insulation, and thus make use not only of their relatively low conductivity, but also of their low heat capacity, which may be less than one-third of that of the normal fireclay brick. The permeability of such refractories is extremely high compared with that of diatomite, but this is not normally of any great consequence and can in any case be remedied by the use of a cement facing.

TABLE XXV  
PROPERTIES OF CLAY INSULATING BRICKS

Code No.	Thermal conductivity at 400°C. (Mean) (British Thermal Units)	Maximum safe temperature °C.	Cold crushing strength ( $9'' \times 4\frac{1}{2}'' \times 3''$ bricks) on end lb. per sq. in.	Porosity per cent.	Bulk density		Permeability through 1 skin c.g.s. units
					g.p.ml.	lb. per cu. ft.	
6	1.61	1300	390	72	0.76	47.4	0.010
11	1.54	> 1200	500	70	0.89	55.2	0.47
14	1.75	1300	370	69	0.79	49.0	3.29
27	2.31	1200	1500	65	0.92	59.8	0.051
28	2.63	> 1300	1190	64	0.88	55.1	0.157
29	2.39	> 1300	430	65	0.85	53.2	1.26
33	1.66	> 1400	400	68	0.80	49.6	1.30
36	2.42	1400	650	58	1.10	68.4	0.135
39	2.55	1400	270	61	1.00	62.5	0.578
41	1.90	1250	740	70	0.74	46.0	0.026
43	2.65	1250	1400	68	0.84	52.5	0.19
45	2.05	1300	470	70	0.80	49.7	0.157
53	2.26	1200	270	63	1.00	62.5	0.15
54	1.49	< 1200	210	62	1.00	62.5	0.33
91	1.62	1350	480	70	0.77	48.2	0.86
92	1.59	1300	216	72	0.71	44.1	1.60
94	1.57	1300	60	77	0.56	35.0	—
95	1.56	1450	360	70	0.70	44.0	7.80
96	2.06	1500	385	68	0.56	35.0	0.64

One of the difficulties often anticipated with hot face insulation is after-contraction. Although this may well prove a problem with the brick having so high an initial porosity, the work of Clements and Vyse suggests that predictions made from test data on blocks heated uniformly may be misleading. They showed that even where such bricks showed appreciable shrinkage in this standard test, they actually showed a slight expansion on the working face when heated from one side in a panel.

### *Silica insulating bricks*

The best porous silica bricks are just as remarkable a product as the highest grade fireclay insulating brick referred to above, but find less application in steelworks mainly because of their much lower thermal shock resistance. They have, however, been used very successfully for the insulation of open-hearth furnace bottoms and have been extensively used in the roofs of the kilns used to fire ordinary silica bricks. Their main limitation is, of course, their high permeability and the relatively small amount of slag required to dissolve away a given thickness of brick. As will be seen from Table XXVI, their conductivity tends to be higher than that of fireclay insulating bricks, as is also their cold crushing strength.

### *Miscellaneous insulating materials*

Insulating materials of other types are grouped together in Table XXVII, although in no way related to one another. Vermiculite, for example, is obtainable either as a loose fill or in brick form, the porosity in each case being just over 1 B.Th.U. at 400°C. (mean). The maximum safe temperature of this material is appreciably higher than that of diatomite, either in loose fill or brick form, but the crushing strength of bricks tends to be very low—only of the order of 100 lb. per sq. in.

Asbestos-base bricks have a particularly low thermal conductivity—of the order of 0.6, but again are incapable of carrying an appreciable load. Other filling materials include rock wool and blast-furnace slag. Both have good insulating properties and are particularly useful for packing between refractory linings and a steelcase, since they can absorb a considerable amount of expansion, though admittedly with loss of insulating value.

Bricks made from sillimanite, kyanite, or batches with alumina contents as high as 75 per cent., are also available. The properties of selected, but not necessarily typical samples are given in Table XVII.

TABLE XXVI  
PROPERTIES OF SILICA INSULATING BRICKS

Code No.	Thermal conductivity at 400°C. (Mean) (British Thermal Units)	Maximum safe temperature °C.	Cold crushing strength ( $9" \times 4\frac{1}{2}" \times 3"$ bricks) on end lb. per sq. in.	Porosity per cent.	Bulk density		Permeability through 1 skin c.g.s. units
					g.p.ml.	lb. per cu. ft.	
18	3.10	> 1300	1140	56	1.04	65.0	1.78
30	3.35	> 1200	580	55	1.00	62.5	1.51
31	1.80	> 1200	330	72	0.69	43.3	2.81
57	2.75	> 1400	590	65	0.83	52.0	0.09

TABLE XXVII  
PROPERTIES OF MISCELLANEOUS INSULATING BRICKS

Code No.	Base	Thermal conductivity at 400°C. (Mean) (British Thermal Units)	Maximum safe temperature °C.	Cold crushing strength ( $9'' \times 4\frac{1}{2}'' \times 3''$ bricks) on end lb. per sq. in.	Porosity per cent.	Bulk density		Permeability through 1 skin c.g.s. units
						g.p.ml.	lb. per cu. ft.	
3B*	Asbestos	0.59	800	60	81	0.41	25.4	0.011
5B	Calcined diatomite	1.30	1200-1300	150	73	0.63	39.3	0.23
13C	Rock wool	0.75	—	—	—	0.20	12.5	—
15C	Vermiculite	1.19	> 1000	—	—	0.15	9.3	—
16C	"	1.10	—	—	—	0.22	13.6	—
23C	Blast furnace slag	1.07	900	—	—	0.87	54.0	—
34B	Kyanite	1.27	> 1250	280	78	0.66	41.2	1.1
75B	Vermiculite	1.28	1100	120	82	0.51	32.0	2.73
66B	75% Alumina	2.95	1450	475	65	1.02	63.5	1.21

\*B=Brick, C=Cement or aggregate.

## CEMENTS, PLASTERS, CONCRETES AND COATINGS

### CEMENTS AND PLASTERS

Insulating bricks, like their refractory counterparts, are frequently laid with cements of essentially similar composition. Thus various diatomite-base cements are made for use with diatomite bricks. A little clay or other plasticiser is, however, often added. As this is liable to promote shrinkage, joints should be made as thin as possible, and dispensed with completely where well-shaped insulating bricks are set between an inner wall and an outer wall or steelcase. There still seems to be some difference of opinion as to whether insulating bricks should be tied-in with refractory bricks. True their expansion properties are different, but on the other hand the insulating bricks are very flexible and can, therefore, accommodate some strain. Certain insulating materials, notably vermiculite and diatomite, are used in plaster form, vermiculite often being plasticised by the addition of bentonite and admixed with a small amount of fibrous material to give some tensile strength. Such materials can be plastered or spread on the outside of brickwork, *e.g.*, checker chamber walls, and where necessary can be kept in position by using nails, driven into the joints of the brickwork, as reinforcing.

### CONCRETE

Most insulating concretes consist of mixtures of ciment fondu (or its American equivalent lumnite cement) with an aggregate consisting of crushed and graded insulating brick—normally diatomite, expanded fireclay or vermiculite—the type of aggregate and the proportion of cement and water being adjusted according to the purpose for which the concrete is required. Such concretes can be cast into large monolithic shapes, thus minimising the heat loss through joints. Where necessary they can be cast *in situ* and the cost of moulding or cutting of expensive special shapes thus avoided. Structures having an insulating concrete back and a refractory concrete face can also be employed. In general such materials are not used above 1000°C., though with special aggregates they can be employed up to 1300°C. Their conductivity is still low, though higher than that of the corresponding insulating brick. Their strength, although not equal to that of refractory concrete, is considerably greater than that of ordinary brick insulation. To get maximum strength, the aggregate should be soaked with water before use and kept wet for 24 hours before drying out. Faster rates can be employed but are liable to lead to weaker structures and possibly cracking.

The proportions and maximum size of aggregate vary considerably, but with calcined diatomite the aggregate may be  $\frac{1}{4}$  in. down and used in the proportion of 4 volumes of insulation to 1 of cement. Similar ratios are employed for vermiculite granules. Any increase in the amount of cement employed yields, as might be expected, a product of greater mechanical strength but lower insulating value.

#### COATINGS

For some purposes, *e.g.*, where it is desired to prevent the entry of dust laden gases or tar, a sealing coat may be employed on the working face of the insulation. This not only prevents permeation of gases but substantially increases the resistance to abrasion. The use of such coatings is, for example, recommended with certain reheating furnaces, or where porous fireclay bricks are employed on the hot face of producer gas mains. Certain of the high alumina cements referred to in Chapter VI can be used for this purpose. Some of them are readily applied, adhere well and dry and fire without crazing or flaking. After prolonged use, however, they do tend to come away from the surface, often bringing with them a thin layer of brick. Certain suppliers recommend the use of a preliminary coat, for example of finely ground sillimanite cement, followed by a second coat of coarser material. Mixtures of sillimanite with chrome ore or chromic oxide have been found to give useful protection to high temperature insulation, where the furnace gases contain iron oxide.

#### APPLICATIONS

##### (a) THEORETICAL

The scientific application of insulation on furnaces demands a grasp of heat flow theory, but fortunately simplified accounts of such theory and its application to specific problems are available. Thus an excellent summary of the factors controlling heat flow by conduction has been given by J. B. Austin in the A.S.T.M. Symposium on Thermal Insulating Materials. The definition of thermal conductivity is given by the coefficient  $K$  in the equation:

$$\frac{dq}{dz} = -KA \, dt/dx$$

where  $dq$  is the quantity of heating passing in the time  $dt$  between 2 parallel isothermal planes of area  $A$  at a distance  $dx$  under the temperature difference  $dt$ .

For most purposes, the integrated equation:

$$q = \frac{A}{x} K (t_1 - t_2)$$

is employed, but it should be noted that this takes no account of any variation of  $K$  with temperature. This difficulty is usually overcome in practice by assuming a value of  $K$  equal to that of the brick at the mean temperature under consideration. This procedure is satisfactory provided the conductivity-temperature relation is essentially linear, when the conductivity at a mean temperature of say,  $400^\circ\text{C}$ ., is the same whether the temperature drop is from  $600^\circ$  to  $200^\circ\text{C}$ . or only from  $402^\circ$  to  $398^\circ\text{C}$ .

The value of  $K$  depends on the units chosen. These vary widely and are frequently confused. The two main standards, however, are as follows:

British Thermal Units: B.Th.U./sq. ft./hr./ $^\circ\text{F}$ . diff./in. thickness.

Centimetre-Gram-Sec.: Cals/sq. cm./sec./ $^\circ\text{C}$ . diff./cm. thickness.

If the result is expressed in C.G.S. units of the type given above, it must be multiplied by 2903 to convert to B.Th.U.

Austin lists the factors which might have an influence on  $K$ :

A. *Chemical composition*

- (1). Influence of molecular structure in pure compounds
- (2). Influence of impurity in solid solution

B. *Physical texture*

- (1). Porosity
  - (a) Total void space
  - (b) Size and shape of pores
  - (c) Size and shape of grains
- (2). Presence of a vitreous or liquid phase
  - (a) Total amount of phase
  - (b) Distribution of phase
- (3). Development of ceramic bond and sintering
  - (a) Influence of time of firing
  - (b) Influence of temperature of firing
- (4). Anisotropy and directional effects

C. *Temperature*

D. *Pressure, stress or strain*

E. *Heat flow*

According to Debye, heat is transferred in solids not only from molecule to molecule, as in gases, but also by conduction of the crystal



lattice as a whole. He considered such solids to be a system of coupled resonators, in which long wave oscillations are superposed on the atomic or molecular motions. Any distortion of the lattice, such as occurs when say iron oxide is added to magnesia, will tend to decrease the conductivity by interfering with these waves. The theory is interesting in connection with refractories, since it suggests that oxides of simple structure, *e.g.*, magnesia, are likely to have a higher conductivity than more complex materials, such as forsterite and mullite.

It must be remembered, however, that the molecular structure is only one factor, and that even high conductivity materials like magnesia can be used as insulators provided they contain sufficient pore space. Such pores may be present as interspaces between crystals, or as micropores in the grains themselves—see the low specific gravity of lightly calcined magnesia. Provided the pore space is small the absolute size of pore does not have a marked effect on conductivity, provided the total porosity remains constant. Foamed refractories containing larger pores, however, tend to show a higher conductivity than bricks of similar porosity containing small pores, apparently because heat transfer occurs not only by conduction but by radiation across the pores themselves.

According to Barrett, the contribution by convection within the pores is negligible at pore diameters of up to 0.1 in. at room temperature and up to 0.3 in. at furnace temperatures. Heat can also be transferred by transpiration of gases under a pressure difference through a permeable refractory wall. The amount of heat carried in this way is, however, normally small and is more likely to occur through joints than through the bricks themselves. It can be reduced by the use of sealing coats on the inside or outside of the wall.

The effect of porosity on conductivity was well illustrated by Austin for diaspore (*see* fig. 134) and silica bricks (*see* fig. 135). So consistent is the variation for a given type or product that the conductivity can frequently be estimated within fairly close limits, provided the porosity is known. This is only true, however, of small variations within a given practice. Thus unfired bricks have a much lower conductivity than hard-fired bricks, due to the poor intergranular contact. Austin, for example, showed that the conductivity of an unfired brick rose from 13.05 B.Th.U. before firing to 20.3 after firing for 24 hours at 1350°C. and to 24.6 B.Th.U. after a longer period at the same temperature. Similar observations have been made with dolomite bricks, where the unfired brick, as used for example in ladle trials, has a markedly lower conductivity than the fired product as used in furnace hearths. This difference between the conductivity of fired and unfired is particularly

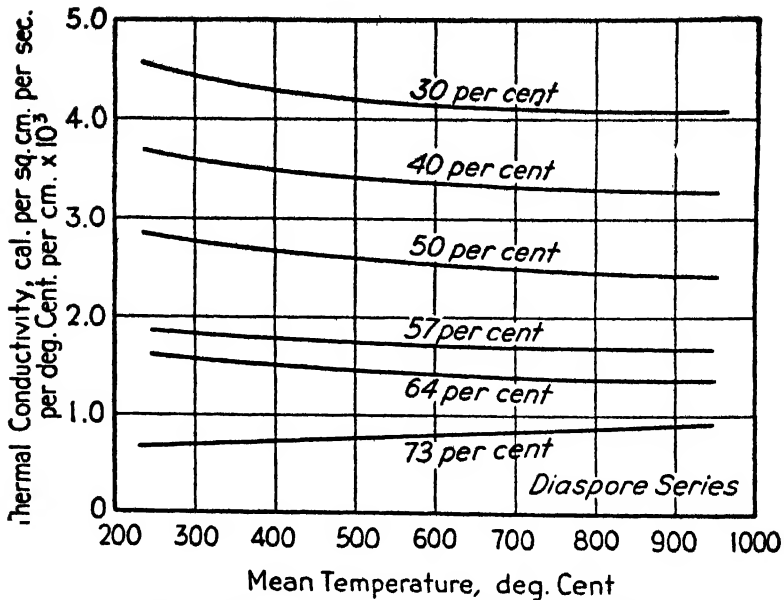


Fig. 134. Effect of porosity on the thermal conductivity of diaspore bricks (after J. B. Austin).

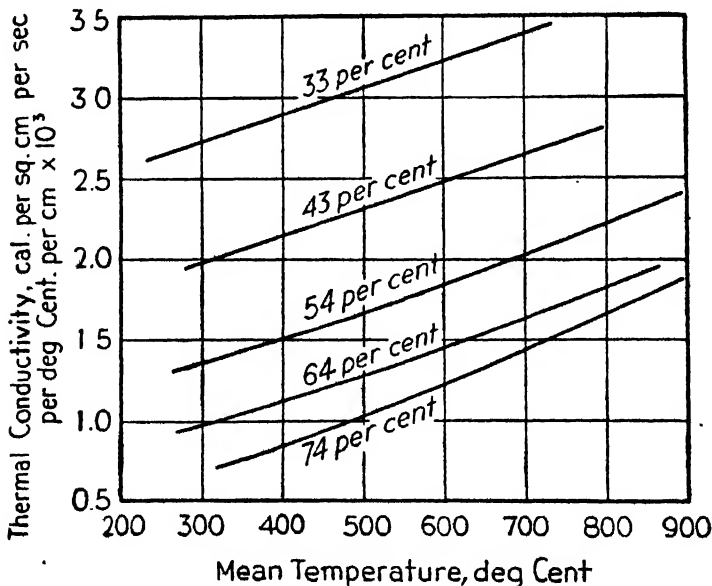


Fig. 135. Effect of porosity on the thermal conductivity of silica bricks (after J. B. Austin).

important with monolithic linings, where only the working face receives the full heat. If for any reason, *e.g.*, an unusually high flux content, sintering occurs throughout an induction furnace lining, then the cooling water temperature rises rapidly and steelmaking becomes less efficient. Similar considerations apply to freshly rammed dolomite hearths, where the initial depth of sintering is small.

#### (b) PRACTICAL

The refractories engineer generally short-circuits the laborious calculations required by a fundamental approach to heat losses, by making use of heat loss curves for equivalent firebrick thicknesses and known inside temperatures. The fundamental assumption behind such curves is that *for equilibrium*, which is often not obtained, the heat loss from a given area of surface of brickwork to the surroundings is equal to the heat which passes in the same time through an equal area of brickwork at any depth within the wall, the outer and inner areas being planes perpendicular to a common axis.

If the outside temperature were known before the calculations started then the average temperature of the brick could be calculated and the conductivity figure for the mean temperature employed. The calculation of the heat loss would then be a simple application of the conductivity equation given in section (a) above. Unfortunately the outside temperature is not known in advance, and hence the standard procedure is to assume a probable outside temperature, calculate the heat transmitted through the brickwork and see how closely this agrees with the heat loss expected from existing relationships for an outside surface at this temperature. If the agreement is not close enough then the assumed outside temperature must be modified and the calculation repeated. The process is liable to be a laborious one, particularly if the wall is composite—involving a number of types of brick of different thickness and thermal conductivity—temperature characteristics. The usual way of simplifying the calculation is to convert the wall to equivalent firebrick thicknesses. Thus if, say, the wall consists of:

	<i>Magnesite</i>	<i>Clay</i>	<i>Insulation</i>
Brick thickness	3 in.	3 in.	3 in.
Thermal conductivity (B.Th.U.)	25	10	1

Then the equivalent firebrick thickness is:

$$3 \text{ in.} \times \frac{10}{25} + 3 \text{ in.} + 3 \text{ in.} \times 10,$$

a total of 34.2 in. Use can then be made of a chart such as that prepared by Etherington, and reproduced as fig. 136, to calculate the heat losses

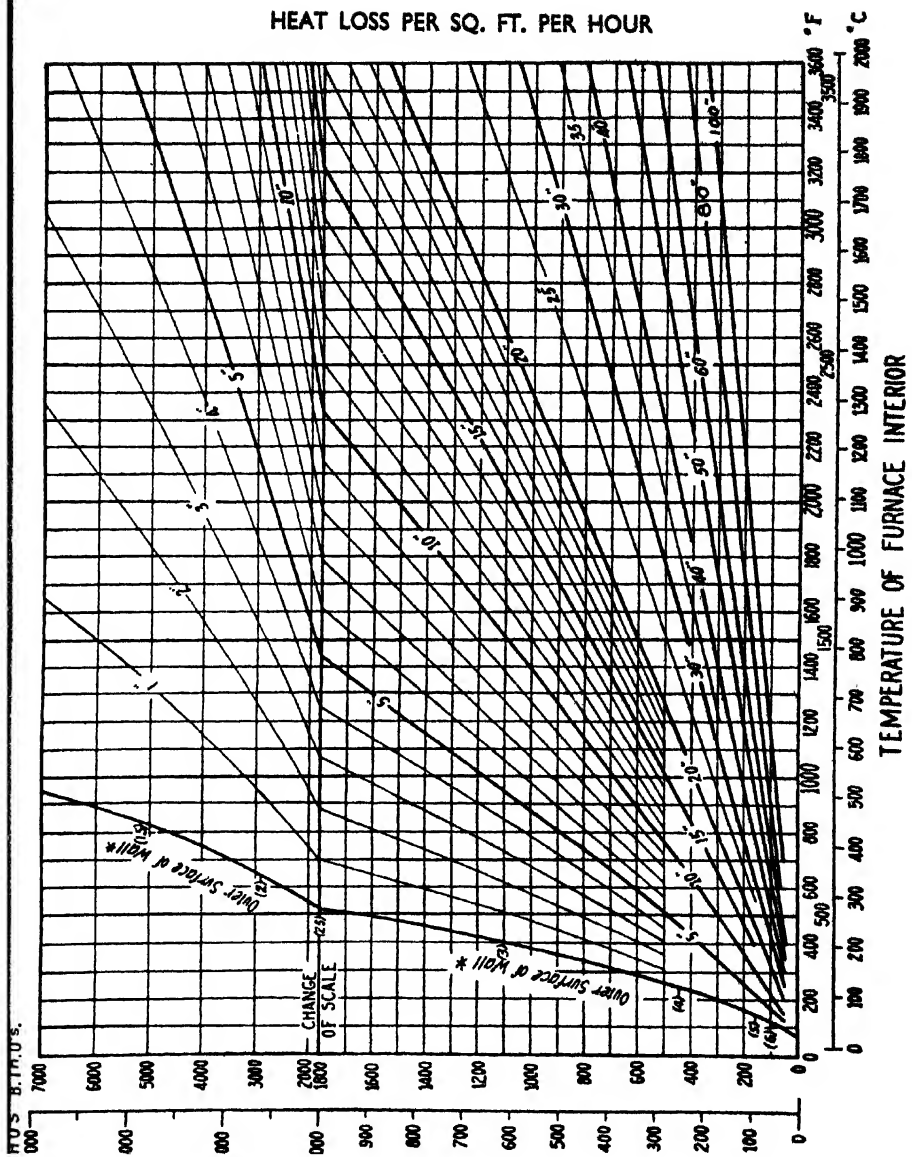


Fig. 196. Chart used for calculating heat losses through furnace walls and interface temperatures. (Reproduced from "Modern Furnace Technology" by H. Etherington, Charles Griffin & Co. Ltd., London, by permission of author and publishers).

and outside wall temperatures for, say, a 35 in. thickness of fireclay brick with any given inside temperature. The chart shown enables calculations to be made for equivalent thicknesses (standard firebrick = 10) of up to 100 in., and for hot face temperatures varying from 0° to 2000°C.

The use of such a chart is perhaps illustrated by taking a specific example, say a reheating furnace roof, having an equivalent firebrick thickness of 15 in. and an inside temperature of 1200°C. A vertical line is first run from the 1200°C. scale at the bottom, and the position of its intersection with the 15 in. curve determined. A horizontal line is then drawn to the left-hand scale to find the heat loss per sq. ft. per hr., which comes out in this case at 1200 B.Th.U. The intersection of this same horizontal line with the curve marked "outer surface of wall" gives, by vertical projection downwards, the outer wall temperature, *viz.*, 220°C. If the effect of replacing such a roof by one consisting entirely of, say, high temperature insulation of conductivity 2.5 B.Th.U., is desired, then the procedure is repeated, but the  $\frac{10}{2.5} \times 15$  in. = 60 in. equivalent thickness curve is used instead of the 15 in., and the new external temperature and heat loss calculated.

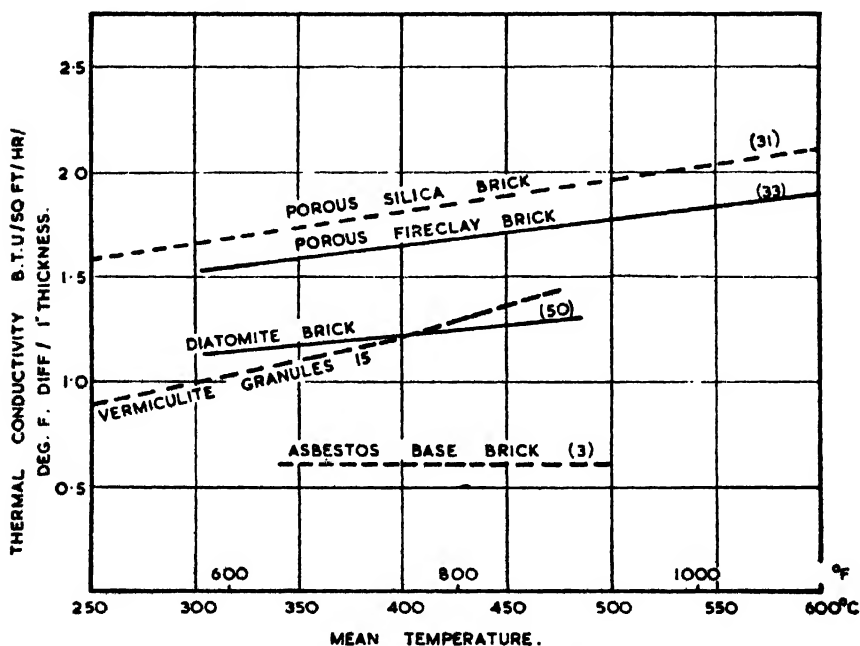


Fig. 197. Thermal conductivity of various insulating materials.

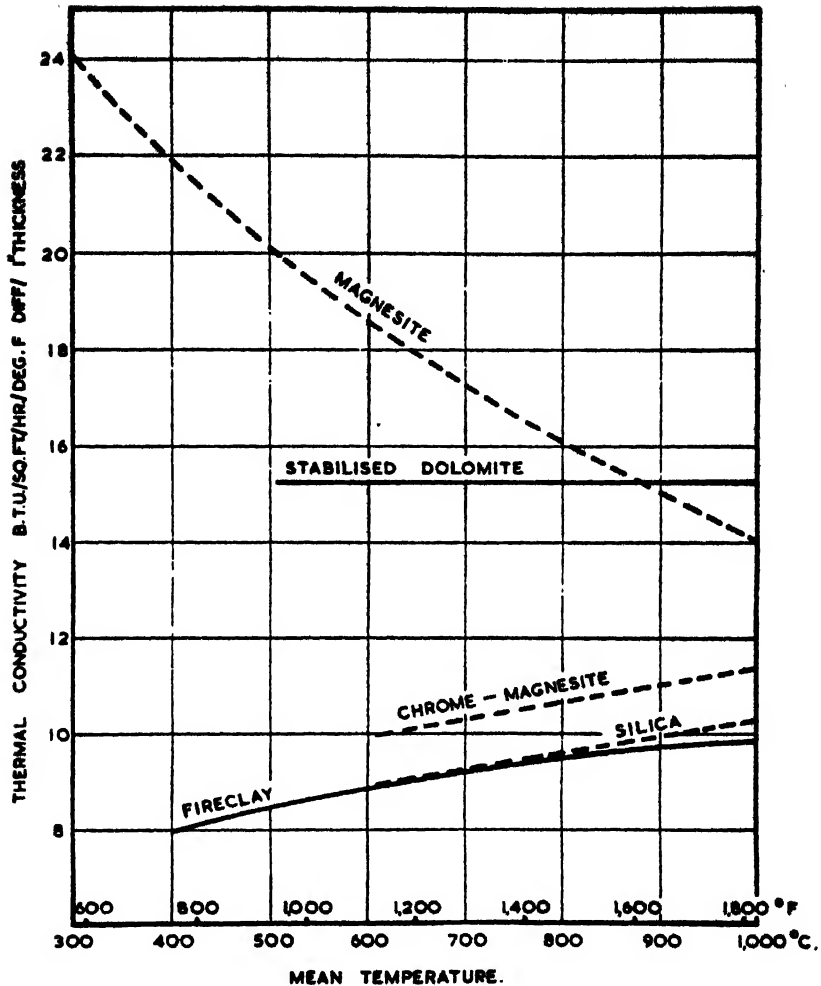


Fig. 138. Thermal conductivity of refractory bricks (magnesite after G. B. Wilkes, fireclay after F. H. Norton).

Strictly speaking the conductivities used should be those of the bricks actually employed to build the furnace, but reasonably accurate estimates can be made by using accepted conductivity data, such as for example are given in figs. 137 and 138. If numerous calculations are required, as might be necessary, for example in working out the most desirable arrangement of insulating bricks for building, say, checker chamber walls, then recourse may usefully be had to some form of slide

rule, such as that designed by J. E. Pluck, and illustrated in fig. 139, p. 315.

This instrument consists essentially of a circular type graph drawn on a rigid base. Temperatures are marked on it radially, and heat losses circumferentially. A line can be seen on the chart giving the heat loss from wall surfaces to still air for various temperatures. A peg is fixed at the radial centre of the chart on which celluloid sheets inscribed with heat loss lines, corresponding to different thicknesses of refractory, can be fitted and rotated. As an example of its use: let us assume that we are concerned with a 9 in. silica brick wall with a temperature inside the furnace of  $1440^{\circ}\text{C}.$ , then the celluloid sheet, with the 9 in. silica brick line on it, is placed in position and rotated until this line intersects the "zero heat" line on the left at  $1440^{\circ}\text{C}.$  (as photographed in fig. 139). If now the point is noted where the 9 in. line crosses the "heat lost from outside wall surface to air" line, then the outside temperature ( $300^{\circ}\text{C}.$ ), and the heat loss (2380 B.Th.U.) can be immediately read off. If the conditions change, *e.g.*, the temperature drops to  $500^{\circ}\text{C}.$ , or a  $4\frac{1}{2}$  in. brick is substituted for the 9 in., it is literally a matter of seconds to get the new outside temperature and heat loss figures.

Where two qualities of brick are concerned, two transparent charts are used, the second being superimposed on the first, but turned over from left to right. To get equilibrium conditions the first sheet is set in the normal way, but the second is swivelled until the temperature at which the radial line from the intersection of the two superimposed curves cuts the "heat loss from the outside wall line" is the same as that at which the curve for the outer material hits the "zero heat" line. This is simply a graphical method of taking account of the difference in conductivity of the two materials and the fact that the heat transmitted through each section must be equal, for equilibrium, to that lost from the outside wall in the same time. The method can be further extended to include three or more materials, though the operation begins to be confusing for the amateur, after the second stage is passed. If use is to be made of a brick having very different characteristics, then a new celluloid sheet can be produced, and used in conjunction with those already available for the standard materials.

The importance of such calculations lies not only in being able to estimate the probable heat saving as a result of insulation, but in being able to say whether an insulating brick in a composite wall is likely to be above its maximum safe temperature. Thus in open-hearth checker chambers it is usually necessary to change from low temperature to high temperature insulation about two-thirds of the way up the wall.

Such experience as has been gained with the application of these rapid methods of estimating both heat loss and interface temperatures suggests that they are sufficiently accurate for all normal purposes.

A more rigid mathematical approach would doubtless give slightly greater accuracy, but it must not be forgotten that the errors of calculation are probably negligible compared with those due to the assumptions made, for example the conductivity of the particular bricks, which is likely to change rapidly in service, and the complete omission of any correction for heat lost through joints, steelplates and the like.

The above considerations apply solely to heat loss under equilibrium conditions. In practice, furnaces are often shut off and allowed to cool long before this state is reached. Equally important then is the question of heat capacity. The outside of the furnace may never get hot enough to dissipate much heat, but the mass of brickwork in the furnace structure requires a great deal of heat to get it to temperature, and this is wasted when the furnace is cooled again. If the furnace is merely insulated by placing diatomite bricks on the outside, the heat wasted in this way (*i.e.*, by storage as distinct from transmission) may be increased, since the average temperature of the heavy firebrick is raised and so, therefore, is the amount of heat stored in the furnace structure when top temperature is reached.

It is for this reason, together with the saving in time associated with quicker heating and cooling, that many steelplants now use hot face insulation, *i.e.*, porous bricks, on the inside of their reheating furnaces. Such a lining not only resists heat flow but has a far lower heat capacity than one made from solid refractories. The construction of furnaces from such friable materials was thought to be extremely risky, but experience shows that provided they are not subjected to substantial slag attack, impact or vibration, they stand up very well, and may lead to considerable fuel savings. Even in furnaces where the conditions are less favourable, trials (at least of panels) are worth while, particularly if accurate fuel records can be kept and savings balanced against probable increases in brick costs. Incidentally the light weight of such structures simplifies furnace construction and may even enable walls to be partially prefabricated, the suspended bricks being attached as a panel to the furnace frame. Given a refractory of sufficient slag resistance, similar techniques might even be applicable, say, to open-hearth furnace roofs, but with present materials the trend is in the opposite direction, *i.e.*, towards bricks of lower and lower porosity, in order to achieve longer roof life.

Commercial literature is often rather optimistic regarding the savings to be expected from insulation, but there is no doubt that with



many furnaces fuel economies of the order of 10 to 20 per cent. can be achieved. What must be remembered, however, is that such savings cannot be calculated merely from data on heat loss through the walls, since other factors, such as changed temperature distribution in the furnace as a whole are involved. Thus insulation of an open-hearth furnace roof without regard to its effect on the rest of the furnace might lead to a slowing up in production, since average fuel rates might have to be reduced to avoid the risks associated with prolonged overheat. Since fuel consumption per ton of product on most furnaces tends to drop with increased firing rate, the savings due to insulation might well be more than offset.

There is still much room for research on insulation, both on open-hearth and reheating furnaces, but to be useful such studies should include far more data on temperature distribution and on the mechanism of heat transfer than is normally available. No one for example is yet in a position to say at all definitely whether a reduction of, say, 5 per cent. in the heat units escaping through, say, the roof of a furnace, also means a saving of 5 per cent. in fuel, or whether, as many workers suspect, the real saving is substantially higher, since the particular heat concerned is at a high temperature level and would, at least in part, have entered the charge.

#### *Effect of insulation on refractories*

In 1935 Harvey described very vividly the effect of insulation on open-hearth furnace roofs, and gave reasons why insulation might lead to greater refractory life. There is no doubt that given an even roof temperature distribution and automatic roof temperature control, the life obtained from a roof should not be decreased by insulation. In practice, however, temperature differences of as much as 50°C. between one point in a roof and another are quite common and local overheats very likely to occur. Under such conditions an insulated roof is liable to suffer much more than an uninsulated one, since the amount of heat it requires to pick-up before a substantial depth, say 2 in., of the surface becomes overheated, is markedly less. This is probably the real reason why insulation of open-hearth furnace roofs is for the most part often abandoned in plants where fast driving is of first importance and roof life already at an inconveniently low level. On the other hand it is found that parts of some furnace roofs are markedly cooler than others, and can, therefore, be insulated without any serious risk of the roof life decreasing.

With front and back walls insulation is less likely to affect roof life, since basic bricks of high melting point are normally employed, but the

economics are extremely doubtful, mainly because of the short life of some of the parts concerned and the loss of insulating materials that results when they are wrecked. To a casual observer the use of water-cooling and insulation on the same furnace may seem quite illogical, but it should be noted that the purposes are quite different: insulation being used almost always to save heat and water-cooling to reduce the damage to refractories at crucial points in the system.

With open-hearth furnace bottoms, over-insulation can have disastrous effects. British furnaces normally have quite thin hearths, *e.g.*, a 12 in. thickness of doloma monolith resting on 9 in. to 12 in. of basic bricks. Since the sub-hearth is normally built of magnesite or dolomite bricks of high conductivity, the use of any appreciable thickness of insulation below these greatly reduces the heat loss to the pan and raises the depth of temperature penetration in the monolith. Other things being equal, such insulation should increase production by avoiding loss of heat from the metal charge, but if, as with one furnace studied, it results in an extremely soft hearth, and a consequent increase in fettling consumption of 50 per cent., then any heat units kept in the charge are more than offset by the additional units consumed during a long fettling period. In addition, the fettling costs both in time and money can increase quite substantially.

## ANTI-PIPING COMPOUNDS

Although anti-piping compounds are not usually classified under insulation, they definitely function as such, and in addition usually supply combustion heat to the molten metal in the mould. It has long been the custom to attempt some measure of insulation, *e.g.* by throwing ashes, coke dust or even straw on top of the steel, with the idea of reducing cavity or pipe formation. Modern anti-piping materials are, however, much more efficient, in that they insulate because of a high porosity—normally obtained by fine grinding—and because they contain substantial quantities (usually about 60 per cent.) of combustible materials, such as anthracite, graphite, cork or sawdust. That they can have a very marked effect on the extent of piping is illustrated by figs. 140, and 141, p. 316.

## GRADING

Tests on 4 commercial anti-piping compounds, having a carbon base, gave the following results:

<i>B.S.I. sieves</i>	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
	%	%	%	%
On 25 mesh	—	—	1	—
25—72 mesh	—	1	4	—
72—100 mesh	2	1	4	4
100—150 mesh	3	4	10	12
150—200 mesh	15	12	15	9
Through 200 mesh	80	82	66	75

It will be seen that brand A, which was one of the pioneer materials, is extremely fine ground, showing 95 per cent. through 150 mesh.

#### THERMAL CONDUCTIVITY

The conductivity of these powders was obtained by pouring them into a brick-shaped well in the low temperature conductivity apparatus described for use with insulating bricks. It will be seen from the data given below that their conductivity at 500°C. mean is of the same order as that of a diatomite brick, *i.e.*, they are good insulators, quite apart from their ability to yield heat by combustion.

<i>Material</i>	<i>Thermal conductivity B.Th.Units</i>	<i>Packing density</i>	
		<i>g.p. ml</i>	<i>lb./cu.ft.</i>
A	1.38	0.66	41.3
B	1.37	0.65	40.6
C	1.00	0.61	38.1
D	1.07	0.59	36.9

#### MELTING POINT OF ASH

At first, the effect of an anti-piping compound is largely one of insulation, but gradually it begins to burn and because of the insulating blanket above, most of the heat enters the steel. As it burns, the carbonaceous material leaves an ash, and if this too is to have an insulating effect, it should remain as a light-weight skeleton and not melt to form a glass that runs into the pipe. The data obtained on the above anti-piping compounds were:

<i>Material</i>					<i>Melting point of ash</i>
A	..	..	..	..	1315°C.
B	..	..	..	..	1315°C.
C	..	..	..	..	1315°C.
D	..	..	..	..	1360°C.





*Fig. 140. Section through ingot head treated with producer ashes.*



*Fig. 141. Section through ingot head treated with high grade anti-piping compound.*

## CONTENT OF COMBUSTIBLES

This was determined by loss on ignition and was found to be:

<i>Material</i>				<i>Content of combustibles</i>
A	..	..	..	63.8 per cent.
B	..	..	..	68.3 " "
C	..	..	..	50.3 " "
D	..	..	..	58.6 " "

## BURNING RATE

Reasonably slow burning is also desirable, since if the material goes rapidly to ash, it is unlikely that the anti-piping compound will keep the ingot top hot enough for the hour or so needed to minimise piping. Tests should, therefore, be made of weight-loss after firing for definite periods at definite temperatures under reproducible conditions, *e.g.*, in small silica dishes in a large electric furnace at even temperature. Tests made on the above compound showed marked divergences in favour of the materials A and B:

<i>Treatment</i>				<i>Percentage loss of combustibles</i>			
				A	B	C	D
½ hour at	850°C.	..	..	14.4	16.2	30.8	22.9
" "	950°C.	..	..	26.7	28.8	51.4	37.6
" "	1050°C.	..	..	36.4	36.5	68.0	47.1
" "	1150°C.	..	..	51.7	52.0	83.2	62.8
" "	1250°C.	..	..	86.5	87.1	97.5	95.7
4 hours,	1000°C.	..	..	100.0	100.0	100.0	100.0

That the above factors are among those controlling the efficiency of anti-piping compounds is supported by the fact that numerous types of material made up on this assumption have given excellent service in the works. Numerous other materials have been used, *e.g.*, ceramic-coated cork, and carbonaceous materials containing powdered aluminium, but the same principles apply, *i.e.*, heat insulation by fine grinding (or the use of a material like cork that is inherently an insulator due to its porous nature), together with heat generation by oxidation of some constituent.

It should be noted that a material may fulfil all the above requisites and yet be unserviceable because of its tendency to carburise or increase the sulphur in the ingot head. For this reason, trials of anti-piping compounds in the works usually include sectioning of the ingot head and taking of sulphur prints and samples for analysis.

In conclusion a new development in the anti-piping or mould top field should be mentioned, *viz.*, the use of mould tiles, made of combustible material, the use of which greatly reduces the sideways loss of heat through the mould and indeed provides additional heat to reduce the rate of freezing and, therefore, cavity formation.



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## Chapter VIII

# THE BASIC OPEN-HEARTH FURNACE

### ABOVE THE SILL-PLATE LEVEL

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**A**LTHOUGH THE OPEN-HEARTH furnace (fig. 142, p. 325) entered the steelmaking field as a rival to the newly invented Bessemer process and in this role has captured well over half the world's output of steel, the prophets of doom still foretell its early demise, due to its relatively low thermal efficiency and slow operating rate. In suggesting that it will soon be replaced by pneumatic methods such as the L-D oxygen blown converter developed in Austria and the more recently announced rotor processes developed at Domnarvet in Sweden and Oberhausen in Germany they tend to ignore several important considerations, for example:

- (1) The relatively high metallic yield from the open-hearth furnace as compared with the Bessemer.
- (2) The relative inflexibility of pneumatic processes, where the making of special alloy steels would, to say the least, be difficult.
- (3) The precision both as regards temperature control and steel composition that can be achieved in the open-hearth due in part to the relatively long times involved, but also to more complete instrumentation and measurement.

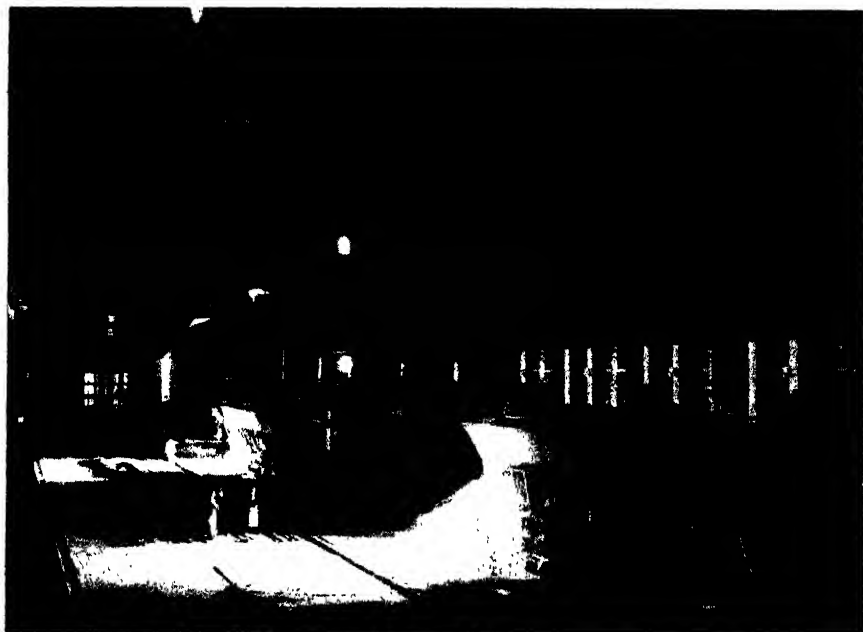
In view of the above, it would appear likely that the open-hearth furnace will be the major method of steelmaking, at least for the lifetime of the present book. For this reason, and also because it probably makes use of a greater variety of refractories than any other industrial furnace, it must receive rather special attention.

The choice of refractories varies appreciably for the different types of open-hearth furnace, *e.g.*, the cold-charged, hot-metal fixed, and hot-metal tilting types, and is best discussed in relation to the different furnace parts. Although the information given mainly relates to cold-charged furnaces of the 80 to 100 ton type, hot-metal fixed furnaces of the 150 ton type, and hot-metal tilting furnaces of approximately 300 ton capacity, similar considerations apply to both smaller and larger furnaces of the same type. As will be seen from the cost data referred to later, the actual life of the refractories varies appreciably both with the type of fuel used and the metallurgical load, but in general the materials used are similar.



The general construction of fixed furnaces fired with producer gas and oil are shown in figs. 143, p. 326 and 144, p. 327. The precise contours do, of course, vary considerably from plant to plant, the particular gas furnace being a semi-venturi type, twin uptake, furnace, and the oil-fired furnace one of the more recent developments in single-uptake design. The illustrations bring out (and indeed exaggerate) the often forgotten fact that nearly two-thirds of the working volume of an open-hearth furnace is below stage, and as such is not even seen by the casual visitor to the melting shop. Emphasis is, however, quite rightly placed on the above-stage section, sometimes referred to as the laboratory of the furnace, since it is here that the charge undergoes the chemical changes needed to convert it to steel and it is also here that the highest temperatures are carried and the greatest control necessary over refractory quality. So much consideration has, however, been given not only to the refractories used in the above-stage parts, but also to the control of temperature and pressure, by means of roof temperature and furnace pressure measuring devices, that the life of these parts has generally kept pace with the increasing driving rate, whilst the below-stage part having been largely ignored is now beginning to show distinct signs of distress. Thus, slag pocket arches that were good for many campaigns, are now subject to collapse, and checker fillings that were once considered adequate for a straight 26 weeks run, are now starting to fail after only 10 to 12 weeks operation. The realisation of this out-of-balance is now leading to increased attention to the below-stage section. This not only includes trials of improved refractories and different methods of suspension, but also stricter temperature control, *e.g.*, over-riding reversal control based on checker top temperature.

For those who are completely unfamiliar with the open-hearth furnace, it should be stated that its essential feature is the combustion above an "open-hearth" of fuel (which may or may not be preheated) in preheated air. The latter is obtained by drawing cold air through the checker chambers on one side, the waste gases exhausting through the corresponding ports at the other end of the furnace and through the slag pockets and checker chambers to the stack. The furnace is reversed at  $\frac{1}{4}$ -hour intervals, and the heat stored in the checkers from the exit gases is returned to the furnaces as preheated air and sometimes also as preheated gas. In the producer gas furnace there are two chambers at each end, a small one to heat the gas and a large one to heat the air. In oil-fired furnaces the twin chambers are sometimes retained to permit of subsequent reconversion to gas-firing, but may be replaced by a single chamber, which is then used to heat air alone.



*Fig. 142. Hot metal addition to 300-ton tilting furnace.*

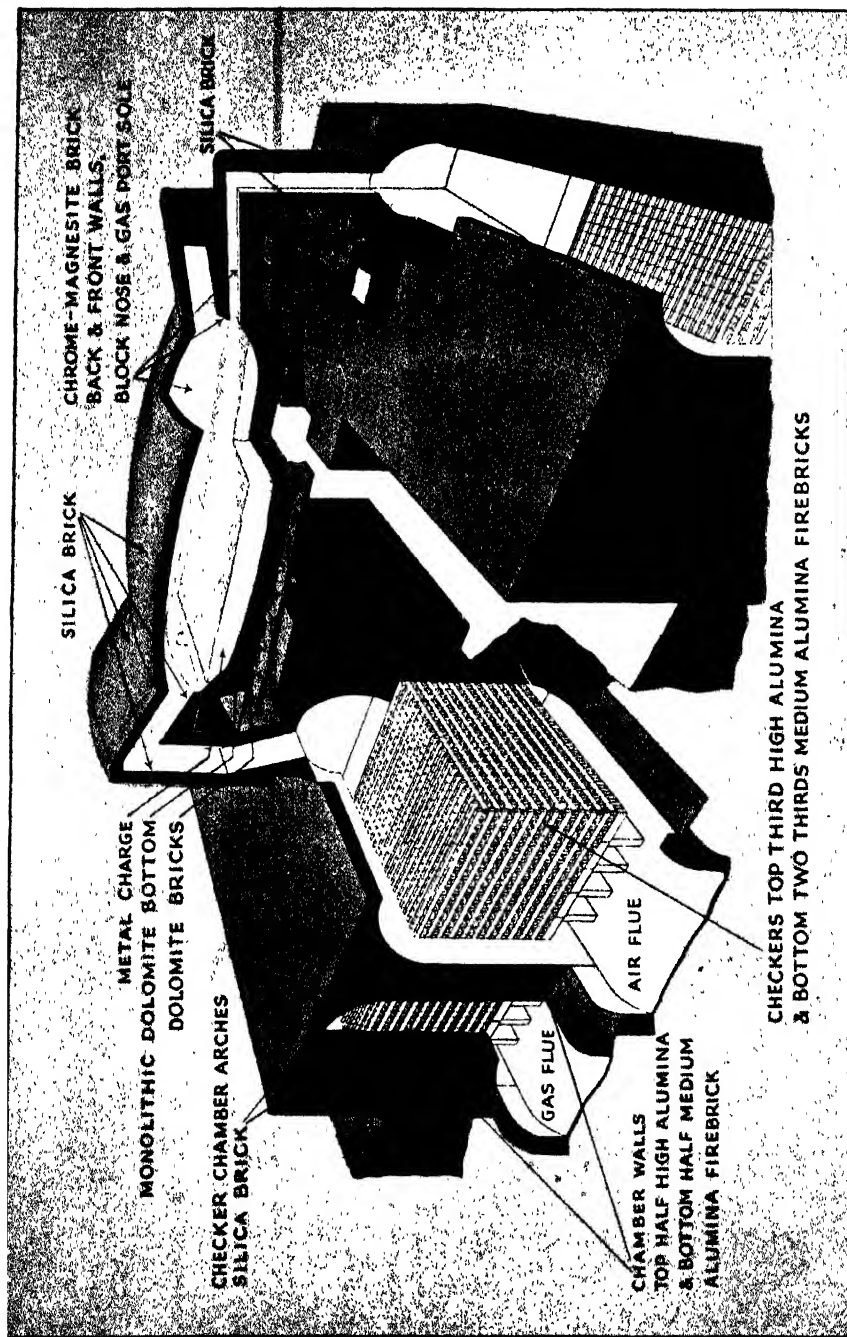


Fig. 143. Producer-gas fired open-hearth furnace (J. Pluck).

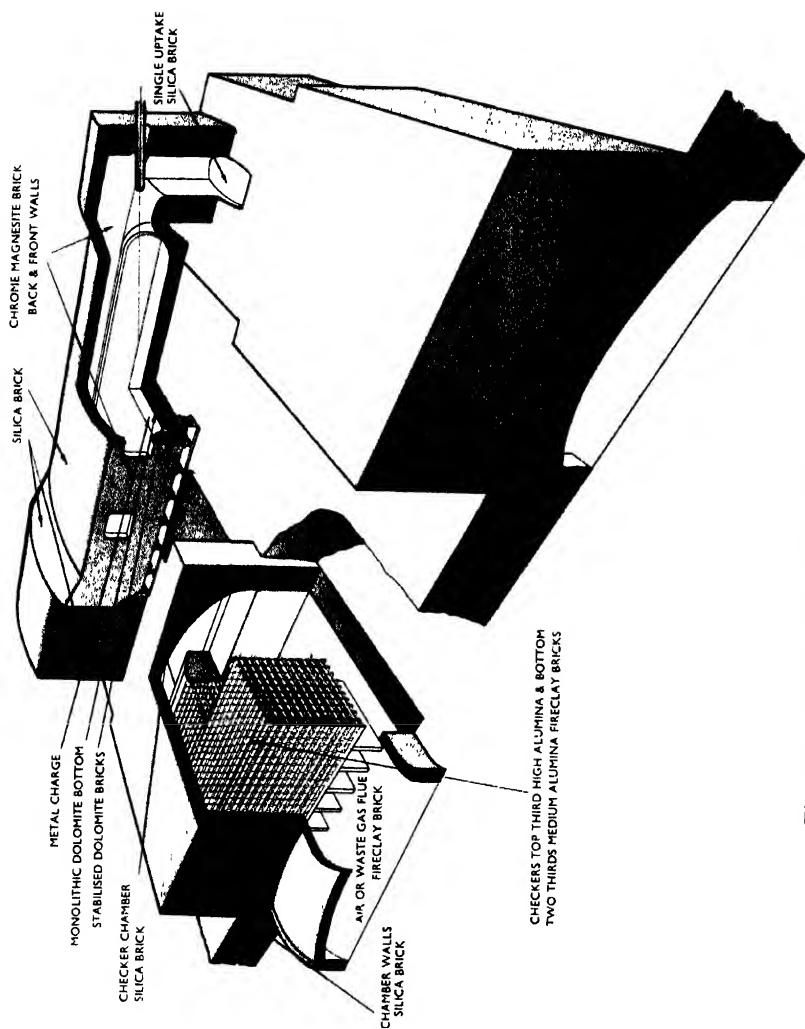


Fig. 144. Oil-fired open-hearth furnace (L. S. Dexter).



The duty imposed on the refractories is in great measure controlled by design, and the consequent flow pattern, mixing pattern and flux droplet deposition pattern within the furnace. This subject is, however, large enough to warrant a book in itself, and will not be dealt with here in any detail. Those who are interested are referred to the references marked with an asterisk in the General section of the bibliography at the end of the present chapter.

## NOMENCLATURE OF THE FURNACE PARTS

Above the stage level the main furnace parts are the roof (both the main section and the ends or ramps), the side walls (back, front and end), the ports (or burners) and the hearth. The latter is the only part in contact with molten steel, but the whole of the inside of the furnace is bombarded by gases rich in iron oxide and sometimes containing lime and basic slag and must, therefore, be capable of resisting these fluxes at temperatures of the order of 1700°C.

The below-stage section may be divided into the slag pockets which act as dust catchers, the fan-tails (rudimentary in most British furnaces) and the checker chambers with their fillings. These in turn are connected through valves and often through a waste-heat boiler to the stack, and in a producer-gas-fired furnace to the producers.

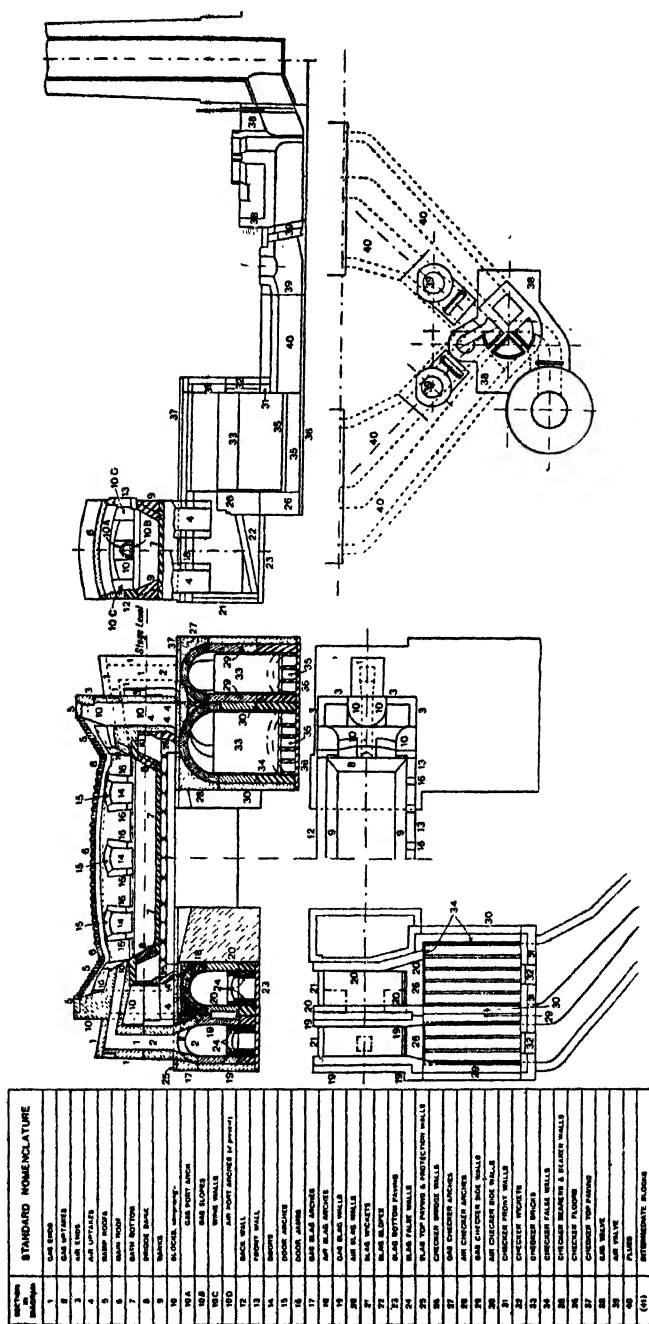
In 1939 the Iron and Steel Institute published a standard nomenclature suggested by the Open Hearth Refractories Joint Panel of Great Britain. This appeared in Special Report No. 26, 1939, and is reproduced here as fig. 145. The terms used for the various parts differ even from one part of Great Britain to another, but are fairly commonly understood both in Great Britain and in America. In the present chapter, sections 1 to 16 will be described with the exception of the hearth (sections 7, 8 and 9), which forms the start of the next chapter. In each case an attempt will be made to summarise the construction employed, the materials used, the life and causes of failure, and possible lines of future development.

What must never be forgotten is that although for convenience a furnace can be divided into parts, and each part considered in an isolated manner, the furnace does in fact work either as a whole or not at all, and should, therefore, as Dr. Sosman pointed out in his address at the opening of the Institute of Silicate Research at Toledo, behave like the . . . "one-hoss shay,

That was built in such a logical way,

It ran a hundred years to a day".

Indeed there are open-hearth men who are so keen to drive hard and repair quickly that they might be prepared to accept even 100 days as



a campaign life provided it meant non-stop production at an even greater output rate.

It is true that hot repairs can often be made to furnaces with relatively little loss of production, but a roof that is capable of lasting, say, a few weeks longer than the corresponding back and front walls, does not pay any particular dividend, since it is not economic to put a furnace back into commission with a roof that can only be guaranteed for a few weeks life. On the other hand a roof or any other part that can be guaranteed to run two campaigns might well be worth more than twice the price, since it reduces shut-down time and only one building cost is involved.

## COSTS

Since the open-hearth furnace accounts for by far the largest part of refractories expenditure in iron and steel works, it is useful to break down a typical set of costs into the principal furnace sections. The figures given below represent rather extreme conditions, the costs for small cold-charged furnaces tending to run high and those for large hot metal furnaces rather low. Hot metal furnaces of the fixed type and intermediate capacity generally show intermediate costs.

<i>Section</i>	<i>80-100 ton</i>	<i>300 ton</i>
	<i>Cold-charged furnace</i> <i>(Materials and labour costs per ton of steel)</i>	<i>Hot-metal tilter</i>
Roof .. .. .	1s. 6d.	1s. 0d.
Front and back walls ..	2s. 6d.	1s. 9d.
Blocks (above stage level) ..	3s. 6d.	1s. 6d.
Bottom and banks .. ..	6d.	3d.
Uptakes (below stage level)	1s. 6d.	3d.
Slag chambers .. .. .	2s. 0d.	3d.
Checker fillings .. .. .	1s. 6d.	3d.
Miscellaneous .. .. .	6d.	—
	<hr/>	<hr/>
	13s. 6d.	5s. 3d.

The big difference in total cost observed is partly the result of furnace size—tapping capacity going up much more rapidly than the brickwork volumes—but is also connected with the type of process operated and the greater amounts of heat that must be supplied to a cold charge. The value of such a breakdown is clearly illustrated by the fact that whereas the above-stage costs are similar, the below-stage costs are almost negligible for the hot metal tilter compared with the cold charged furnaces. The explanation in this particular instance is undoubtedly



that although much work has been done on the maintenance of the above-stage section in this fast driven furnace, the downstairs portions are much as they were in slow working days, and are, therefore, suffering very substantially. Conditions in the hot-metal furnace on the other hand, although quite severe above stage are relatively mild below, the total refractories costs being less than that for the checker fillings in the cold-charged furnace.

Many other factors affect refractories costs, *e.g.*, metallurgical load—extra slag thickness raising the temperatures required to drive the heat into the charge at a given rate—and fuel type which can have a big effect. Thus it has been found that cold-charged furnaces fired with pitch-cresote instead of with fuel oil may show a refractories cost approximately 2/6*d.* per ton, *i.e.*, say, 20 per cent. greater than when oil fired.

Comparison of British and American costs is extremely dangerous, since it involves an assumption regarding rate of exchange. It is, however, interesting to note that if due allowance is made for the difference in brick and labour costs in the two countries, the figure obtained for most American furnaces (about 2 dollars per ton) would fall between those given in the above table.

Elementary arithmetic suffices to show that this is clearly a field where research and control should pay dividends: thus a reduction of roof costs from say 1/6*d.* to 1/- per ton (an increase in roof life of roughly 50 per cent.) is equivalent in a shop making 1,000,000 tons of steel per annum to a saving of £25,000—sufficient to warrant the employment of a score of technicians.

## THE ROOF

*(Sections 5 and 6)*

Although more research has probably been done on open-hearth furnace roofs than on any part of any other furnace it still remains one of the most crucial sections, its life frequently determining the time at which the furnace campaign ends or intermediate repair takes place.

### (a) CONSTRUCTION

#### *Shape*

The shape of an open-hearth furnace roof is an integral and very important part of its aerodynamic design. If for example it rises substantially between knuckles then marked recirculation below the roof is likely to occur, whilst if the central section is lower than the ends recirculation is likely to be slight. That such considerations are vitally

important to the refractory man is shown both by the flow pattern observed in air and water models and by tests on actual furnaces. These all indicate, at least for the furnaces tested, that reverse flow beneath the roof is quite common, at least for the exit half, *i.e.*, that the flame after impinging on the bath and picking up iron oxide runs up the back and front walls, impacts on the roof and then streams backwards towards the active burner. At some balance point, frequently one-third of the distance between the active burner and the non-active one, it meets the incoming air stream and the two combine and re-enter the main jet. Such returning gases deposit substantial quantities of iron-oxide on the roof (sometimes as much as 1 lb. per sq. ft. per hr.) and do so in a manner determined by the flow pattern. Thus, there is generally heavy deposition in the regions adjacent to the back and front wall. The distribution, which is determined of course by many factors apart from roof shape, may lead to comparative deposition rates as high as 40 : 1 between two points on the same roof at the same period during the heat.

The original Siemens furnace was of course fired with producer gas, and use was made of a sloping ramp at each end to drive the air down into the gas stream and thus promote mixing. With the low momenta available in such furnaces, this procedure was essential if substantial quantities of gas were to be burned in the space available, but with driven fuel furnaces, such as the modern oil, tar or combination of fuel types, the momentum needed to mix the air with the fuel is supplied by the high pressure steam or air used initially to atomise the fuel. Such a furnace can operate quite satisfactorily as regards combustion without any knuckle, as indeed did certain producer gas-fired furnaces, *e.g.*, the Maerz. Even so most modern furnaces even though fired with driven fuels show at least a slight knuckle with an intermediate portion that is either flat from end to end or cambered upwards. Very few roofs are built flat from back to front, and the basic design may therefore be considered as a cylindrical section. Clearly the simpler the design the easier it is both to build and to repair.

### *Ribs*

Spotts McDowell has discussed fairly fully the relative merits of plain and ribbed roofs, the main object of the latter being to provide additional strength without any great increase in weight. Roofs built in straight rings without ribbing are now unusual, and even ribbed roofs suffer from certain limitations, *e.g.*, the tendency of a whole course to fall in when an odd brick or two spalls or melts. In the United States 12 in. roofs with 15 in. or 16 in. ribs, 13½ in. and 15 in. roofs with 18 in.

ribs, and 18 in. roofs with 22½ in. ribs, are common practice. In Great Britain on the other hand roofs tend to be thinner, 12 in. roofs with 15 in. ribs being more general. Certain of these are, however, built 15 in. thick with 18 in. ribs in the region adjacent to the back wall.

An interesting new development is the so-called Kreutzer roof, where longitudinal ribs are used in addition to the normal transverse ones. Such a roof is obviously more difficult to build, but possesses the advantages of great stability and ease of fitting filler bricks in the boxes formed by the cross ribs.

### *Bonding*

In Great Britain there is an increasing tendency to use bonded roofs in view of their greater stability. Such a roof takes a slightly longer time to install and calls for more care in construction, but it is considered that with many furnaces this is more than paid for by the ability to run whole areas of the roof very thin without collapse occurring.

### *Suspension*

Some measure of suspension or restraint seems essential with basic bricks, but with silica no obvious advantage accrues and consequently the use of such roofs is most unusual. Even the removal of pressure between the bricks during the heating-up period is a doubtful advantage, since if thermal spalling does occur lateral pressure will help to keep the fragments in position until such time as the roof glazes over and becomes monolithic (*see fig. 146, page 345*). The idea that suspended construction facilitates repair is open to doubt, since the mere presence of a forest of metal hangers makes access to the sore point very difficult. Furthermore, as numerous articles in the A.I.M.E. Proceedings testify, steelmen have become adept at carrying out local roof repairs even on hot furnaces.

With a sprung roof, the skewback channels are normally suspended, and the bricks themselves designed to avoid vertical slip, *e.g.*, by a tongued and grooved arrangement. The importance of maintaining both the condition and alignment of skewback channels cannot be over emphasised. At one time brick shape was so poor that there was little point in ordering bricks with only a slight taper. Such bricks made today either in hydraulic presses or loose-lined moulds are of excellent shape, and roofs can now be built satisfactorily and almost entirely from end-arch bricks, a few wedge bricks being left above the general level during building and subsequently driven home to key up the arch and lift the roof from the wooden or metal centres.

*Thickness*

Most open-hearth furnaces in Great Britain are built with 12 in. bricks, but in the United States heavier roofs are normal. Thus Buell says that the tendency with furnaces of more than 125 tons capacity is towards 18 in. thick roofs with  $22\frac{1}{2}$  in. thick ribs.

*Rise*

There would appear to be a general tendency, at least in Great Britain, for roof rise to be stabilised in the range  $1\frac{1}{2}$  in. to  $1\frac{3}{4}$  in. per foot of chord, though considerably higher rises are occasionally employed. The use of a lower rise, say 1 in. per foot, is not advisable, since it frequently leads to flattening of one section and subsequent collapse due to the roof "breaking its back". Even with hard-fired silica bricks and a normal rise, a roof of 18 ft. span may rise by as much as 6 in. during the heating-up period. To ensure that this rise is evenly distributed, packing by means of brick pillars placed on the roof and end cross-girders, is sometimes employed. According to Buell, the horizontal thrust on the skew back of an open-hearth furnace roof is given by the equation:

$$HF = \frac{\frac{1}{2}W \times C \times K}{8 \times R}$$

where  $HF$  = The horizontal thrust in pounds.

$W$  = The weight of the roof in pounds (3 ft. wide section).

$C$  = The length of the chord in feet.

$R$  = The rise of the roof in feet.

$K$  = A coefficient (2.0).

The coefficient  $K$  is an empirical safety factor included to take account of "thermal stresses developed within the roof, whilst under heat".

This phrase is presumably intended to cover such things as the peculiar pressure distribution associated with keying up, removal of centres and subsequent heating-up of the roof. Thus a freshly keyed up roof may show maximum stress at the bottom, but this is transferred to the top when the centres are removed and returns again to the bottom when the hot face goes through the cristobalite change. It is interesting to note that if the rise is small, the horizontal thrust on the skew back can become very substantial.

*Expansion allowances*

In general the longitudinal expansion in roofs of hard fired silica bricks is approximately equal to the thermal expansion of  $1000^{\circ}\text{C}.$ ,

*viz.*, 1.25 per cent. This allowance is made either as a number of rather wide transverse gaps (parallel to the roof rings), or in a bonded roof by means of spacers, *e.g.*,  $\frac{1}{8}$  in. felts, placed between every other brick in planes perpendicular to the length of the roof and 12 in. apart. No expansion is allowed transversely. Since it is by no means certain that the expansion of individual bricks is transmitted over considerable lengths of roof or side walls, the use of a large number of small gaps is generally preferable. The use of felt has proved advantageous, not only because it burns out at a low temperature to give only a small amount of ash, but because it provides a cushioning effect, particularly during the early stages of heating-up. Laboratory tests suggest that tarred roofing felt, which softens under load at a relatively low temperature, is preferable to either carpet felt, whose thickness is indefinite, or wood, which is liable to burn out too slowly.

### *Cement*

Certain furnace builders still use cement for setting roof blocks, but it is doubtful whether there is any advantage in so doing, provided the bricks are of good shape. If cement is used it should be finely ground and have a refractoriness of the same order as that of the bricks themselves. Special cements are advocated by Kreutzer for the setting of filler bricks in the box-shape openings of this type of roof.

### *Insulation*

All roofs are partly insulated, in as much as they soon become coated with a layer of low conductivity dust. Certain firms consider this so disadvantageous that they deliberately remove it at intervals, whilst others still believe in some measure of deliberate insulation. Given a roof having a perfectly uniform temperature on the inside and completely foolproof temperature control, there is little doubt that insulation would not result in a reduction in roof life and would lead to at least a small fuel saving. If on the other hand an insulated roof becomes overheated then, as has already been mentioned, the damage is likely to be far more than with its uninsulated counterpart. Certain plants recognising that particular parts of their roofs tend to operate at a lower temperature than others, insulate these particular areas, thus obtaining a more uniform wearing of the roof and presumably some fuel saving. The most common method of roof insulation, is the use of expanded vermiculite, which not only has a low conductivity but a sufficiently high maximum safe temperature to enable at least part of it to be recovered for use in subsequent campaigns.

**(b) MATERIALS***Specifications*

When Siemens built his first open-hearth furnace he made it of Stourbridge fireclay bricks, but shortly afterwards (in 1868) Sir William Siemens stated in a paper to the Chemical Society that he had recently tried out a Welsh Dinas brick, consisting of pure silica. This enabled him to work at such high temperatures that a Stourbridge firebrick charged into the same furnace was soon so soft that it could be drawn out into threads. He would doubtless have been surprised to know that nearly 100 years later there was still no universal agreement as to the properties which such silica bricks should possess for use in open-hearth furnace roofs, the only existing specification being the D.I.N. 1088, which can be summarised as follows:

Chemical analysis	..	SiO <sub>2</sub> 94.5 per cent. minimum
		Al <sub>2</sub> O <sub>3</sub> 2.0 per cent. maximum
		CaO 3.5 per cent. maximum
Cone melting point	..	At least Seger cone 32 (1710°C.)
Refractoriness-under-load		Beginning of failure (2 kg./sq. cm.)
		1630°C. (minimum)
Total porosity	.. ..	Less than 25 per cent.
Specific gravity	.. ..	Less than 2.43
Cold crushing strength	..	Over 100 kg./sq. cm.

Although the above specification has proved serviceable, many bricks whose properties fall within it, would still not be acceptable in most British, or for that matter American steelplants. Thus a maximum figure of 2 per cent. Al<sub>2</sub>O<sub>3</sub>, which seemed reasonable 20 years ago, is now considered excessive, 1 per cent or even 0.8 per cent. being preferred. The specific gravity would also be considered far too high for adequate control, bricks in the 2.32–2.34 range being preferred in Great Britain and America, though still not popular in Germany.

*Grading*

Until comparatively recently coarsely graded bricks were preferred for use in open-hearth furnace roofs, doubtless because being usually also soft-fired they possessed a high thermal shock resistance, and were less likely to be damaged during the initial heating up. Given tight control over this stage finer ground bricks are preferred, since they are generally a better shape, glaze more readily and wear more evenly.

*Shape*

The importance of good shape was emphasised by Smith and McKendrick, when they showed by means of special tests that warped

bricks heated up with their concave surfaces together, would rupture very readily. The excellence of modern roof bricks permits of the use of special slow tapered bricks, *e.g.*, 12 in.  $\times$  6 in.  $\times$   $3/2\frac{1}{2}$  in., for the bulk of the roof, with occasional bricks of larger taper to facilitate keying.

### *Other properties*

The significance of melting point, specific gravity, bulk density and refractoriness-under-load data, are discussed in Chapter II, in connection with the properties of silica bricks. It only remains here to stress the desirability of repeated checking on deliveries to make sure that the alumina content is below the desired maximum and that the control of firing as shown by specific gravity results is adequate. Demands as regards bulk density and refractoriness-under-load will depend on the class of material under consideration, but should in both cases be as high as possible. With standard silica roof bricks, bulk densities of more than 1.8 are somewhat unusual, but with special super-duty bricks, *e.g.*, of the silcrete type, values exceeding 1.9 g.p.ml. can be expected. Incidentally the determination of alumina in silica bricks has been greatly facilitated by the work of Irish, Mason and others, who have shown the possibility of using the spectrograph for this purpose. The 8-Hydroxyquinoline method has also proved most useful.

### *Cements*

Where cement is used with silica roof bricks, it is generally made by grinding up used silica bricks or ganister, and adding a small amount of lime or clay as bond. Sulphite lye may also be added to give stickiness. Given reasonable workability, the property most important is high refractoriness, a cone melting point of at least 31 (1690°C.) being desirable.

### (c) LIFE AND CAUSES OF FAILURE

The comparison of roof lives at different plants often results in confusion, partly because the units employed vary greatly. Thus the result may be quoted in heats, weeks, or tons, and the amount of repairs carried out may, or may not, have been included. The Refractories Committee of the American Iron and Steel Institute made a real contribution in this field, when they suggested (*see* A.I.M.E. Open Hearth Proceedings, 1948—H. M. Kraner) that roof life should be reported on a standard basis, the procedure to be adopted being summarised as follows:

- (1) Comparison shall be made on the basis of unit roofs plus all repairs made.
- (2) Repairs shall be reduced to terms of unit roofs or fractions thereof.
- (3) A unit roof is a  $13\frac{1}{2}$  in. thick cover of any open-hearth from knuckle to knuckle, or its equivalent in 9 in. equivalents ( $9 \text{ in.} \times 4\frac{1}{2} \text{ in.} \times 2\frac{1}{2} \text{ in. brick, } 101.25 \text{ cu. in.}$ ).
- (4) A unit roof need not be a uniform-thickness roof. It may consist of several thickness sections, such as rib roofs, heavy shoulder sections, or other combinations. The volume of the unit roof for any furnace is easily calculated at 9 in. equivalents or volume and this value can be used as the divisor in determining the number of unit roofs used during the campaign.

Attempts have been made to introduce a similar system in Great Britain, and although no official standard exists, a considerable number of firms do now report their roof lives on a unit basis. It should, however, be noted, that for convenience the British life is expressed in terms of a 12 in. instead of a  $13\frac{1}{2}$  in. cover.

Expressed in these terms unit roof life in the United States ( $13\frac{1}{2}$  in. basis) usually runs at about 100 heats, and in Great Britain (on the 12 in. basis) at the rather higher figure of 150 to 200 heats.

The extraordinary differences in roof life between plants are gradually being reduced. Whereas at one time some firms only got 5 weeks and others 50 weeks, there is a tendency to increase the shorter lives by better operating control and to decrease the longer ones by faster driving. As a result a figure of 13 weeks, or, say, 4 roofs per year, is becoming increasingly common.

A full discussion of the factors controlling roof life on open-hearth furnaces, would occupy a book rather than a few pages. The statement given below must, therefore, be highly selective and intended to convey the type of problem involved rather than answer specific problems.

### *Design*

A great deal of information is now available regarding the way in which design affects roof life. This has arisen primarily from the study of flow in  $\frac{1}{4}$ th scale water models, followed up by comparative tests on actual furnaces, using such things as burning wood blocks to trace the flow of gases. The results obtained from the early model work were so unorthodox as to lead to doubts as to whether they could be true of actual furnaces. Numerous cross-checks, however, indicate that the story as told by the water model is substantially true, and that complex



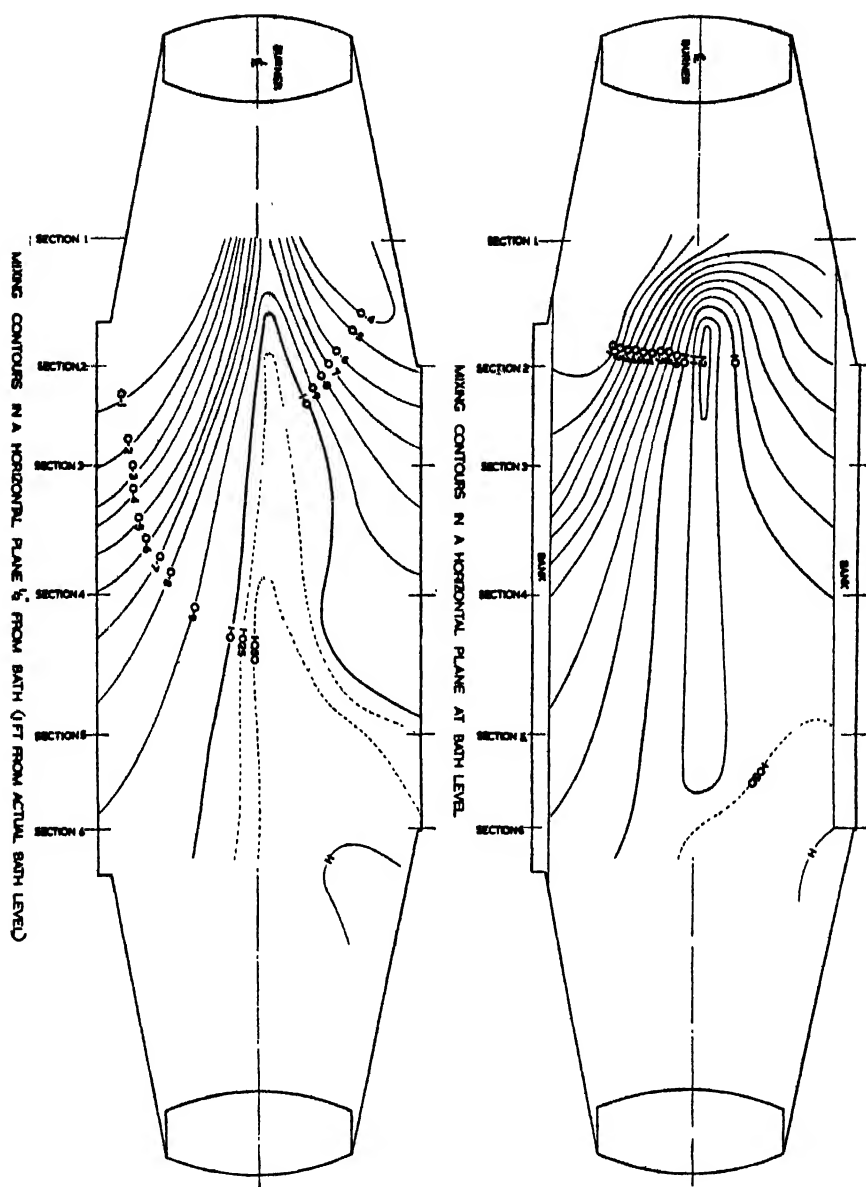
flow patterns, such as that shown in fig. 147 do in fact occur. Indeed this particular pattern is a relatively simple type and as such facilitates description. It will be seen that in this venturi type producer gas-fired furnace, the air forced downwards by the ramp roof combines with the jet of gas from the relatively large port to form a flame, which impacts on the bath and then splashes in all directions but particularly forwards and sideways. Much of the central stream after passing over the bath leaves by the gas and air downtakes at the other end. Substantial parts of the burning gases, however, run up the back and front walls, where they impact on the roof. They then turn in towards the longitudinal centreline. A large part returns towards the active gas port and is re-entrained by the flame.

This recirculation, which is characteristic of all the furnaces so far examined, is undoubtedly responsible for much of the observed roof wear. It explains, for example, the characteristic guttering of the roof in a region adjacent to the back wall (*see* fig. 148, p. 346) and generally to a less extent in a region adjacent to the front wall, these being the regions where the gases having lifted iron-oxide fume from the bath are forced to change direction suddenly and thus shed a substantial part of their fluxes.

Thus, although to the casual observer the whole of the furnace may appear bathed in fume and, therefore, likely to wear at a relatively uniform rate, there are in fact areas where deposition is highly concentrated. That this actually is so can be readily shown (*see* McInerney) by placing a dozen or more roof bricks containing holes through them at strategic points on the roof and carrying out a series of trials at various stages during the heat, in which sintered alumina thermocouple sheaths are pushed an inch or two into the furnace for a definite period and then withdrawn and the iron oxide pick-up determined by chemical analysis. It will be found that the pick-up on test-pieces exposed for identical periods of the heat will vary by as much as 40 : 1 (fig. 149 p. 346). If thermocouples are inserted in these probes it will also be found that substantial temperature differences exist, which in turn are often explainable in terms of model work. For this purpose mixing type models, *e.g.*, those in which the gas is represented by hot air, at, say 300°C., and the air by cold air, are employed. Distribution patterns such as that shown in fig. 150, are then obtained by means of traversing thermocouples. The particular illustration shows that even with a symmetrical furnace chamber the mixing pattern is anything but symmetrical, tending to be more rapid in this case on the front wall than on the back wall, due to the asymmetrical way in which the air enters the furnace. This in turn is the result of a straight run from



Fig. 147. Flow pattern in model of gas-fired venturi type open-hearth furnace.



the checkers over the bridge-wall, impact on the pit-side wall of the uptake and consequent high velocity flow on the pit-side compared with the charge-side.

Although the fundamental answer to such irregularities lies in complete redesign, some assistance may be gained by remedial measures, such as turning the burner a few degrees towards the front wall, or displacing it towards the front wall, but keeping it in a direction parallel to the longitudinal axis of the furnace.

### *Brick quality*

It is not long since technologists held the view that little difference in life could be expected between different brands of first quality silica brick, since their melting point as determined by the pyrometric cone method was always so close to that theoretically possible. This complacency was completely destroyed by Hobart Kraner, who did a signal service to the industry, by demonstrating a marked correlation between roof life and alumina content in silica bricks. This theory, which was supported by phase-rule studies, suggested that even a reduction of 0.1 per cent. alumina was worth striving for. The effect of this discovery was reinforced by the introduction by Birch and others in the United States of the so-called super-duty brick, which specified a maximum content of alumina plus alkalis plus titania of 0.5 per cent. Shortly after the publication of this work, the Oughtibridge Silica Firebrick Company, in Great Britain, in co-operation with the United Steel Companies, decided to import a few thousand tons of silcrete from South Africa, having been told that bricks made from this material gave markedly longer lives than the best British and American bricks then available. The results of split roof tests of the type suggested by Kraner were truly sensational, being illustrated quite fairly by the photograph shown as fig. 151, p. 347. It will be seen that whereas the silcrete half of this particular roof was virtually untouched after 98 casts, the other half was badly worn although built from what at that time were considered to be first-class silica bricks. Although subsequent trials using complete silcrete roofs did not give the improvement in life expected—presumably because the melter always tends to carry as high a temperature as he dare and thus get greater output—great benefits have been gained by the use of this material as panels adjacent to the back and front walls to reduce guttering. In some furnaces the necessary improvement has been obtained by the use of silcrete in panel form, as shown in fig. 152, p. 348, where the contrast between the projecting ribs and the adjacent bricks leaves no room for doubt.

Examination of the factors responsible for the remarkable success of this South African material led to certain new clues, as follows:

- (1) High bulk density is as important as low alumina content.
- (2) Titania—at least in the form present in silcrete—cannot be considered as deleterious as the American super-duty patent would suggest. Thus a typical analysis of a silcrete brick shows only 94.7 per cent. silica, an alumina content of 0.34 per cent. and titania content of 1.77 per cent.

The result of all this work has been that manufacturers in Great Britain and in the United States, have made determined attempts to reduce the alumina content of normal quality bricks, *e.g.*, by more careful selection of rock, by washing of raw materials, and by the use of sand in the fine fractions, whilst several low alumina—high bulk density silica bricks, competitive with imported material, have been produced from British resources. There is no doubt that this advance in thinking has led to a substantial improvement in roof life.

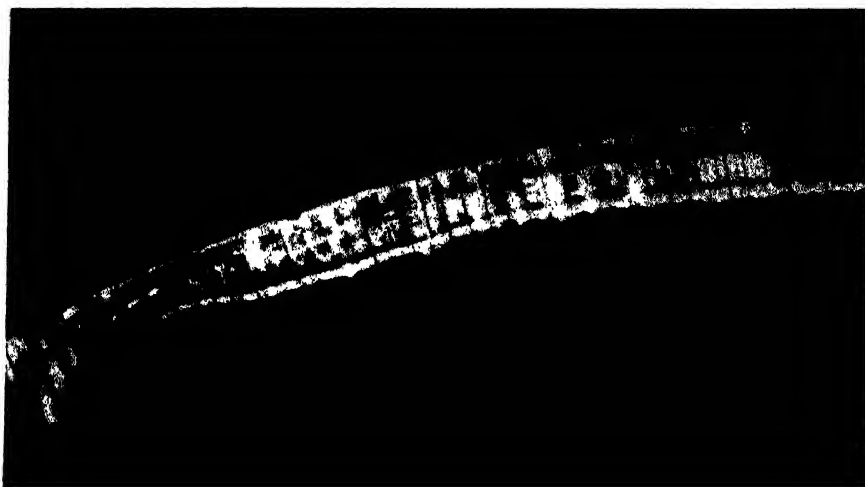
### *Fuel*

Many plants have observed a change in roof life as a result of changing from one fuel to another, even where every attempt has been made to maintain both combustion and metallurgical control at a constant level. It would appear that the differences are due, at least in large measure, to two factors, *viz.*, momentum in the fuel jet and what might be described as specific fuming tendency. It might be expected that the use, for example, of driven fuels, where the velocity at the nozzle may exceed that of sound, would result in greater velocities both at the bath and in subsequent flow over the refractories. Further that these high velocities would almost inevitably carry larger amounts of fluxes to the bricks. That this is, in fact, the position is illustrated by the increased wear observed in the exit end of the furnace roof, where steam/oil ratios have been raised, or high pressure cold oxygen jets used for flame enrichment. In both cases guttering, normally limited to the tap-hole region, has spread outwards into the ramps and tended to become excessive, in spite of roof temperature control.

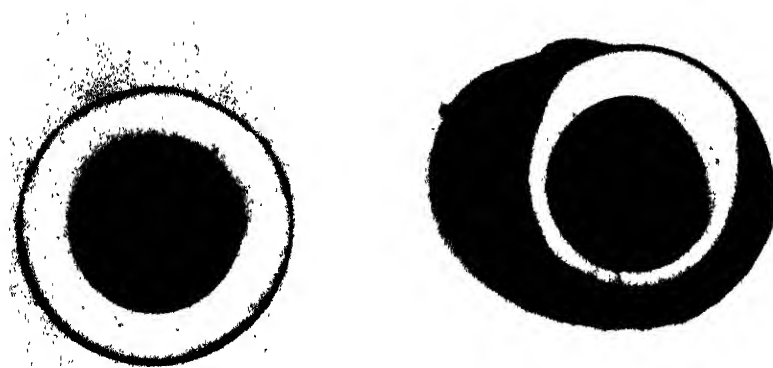
Users of tar or pitch-creosote are well aware of the marked increase in carry-over of dust to checkers, which occurs with such fuels when compared with oil. Since fuming may be considerably reduced by, for example the use of oxygen for combustion, or by higher steam/oil ratios, the suggestion made by certain German workers, that fuming is associated with the presence of carbon from the fuel, seems a reasonable one. Whether, as they suggest, an unstable iron-carbonyl is formed due to the impact of carbon-rich fuel on the scrap, is as yet unproved.



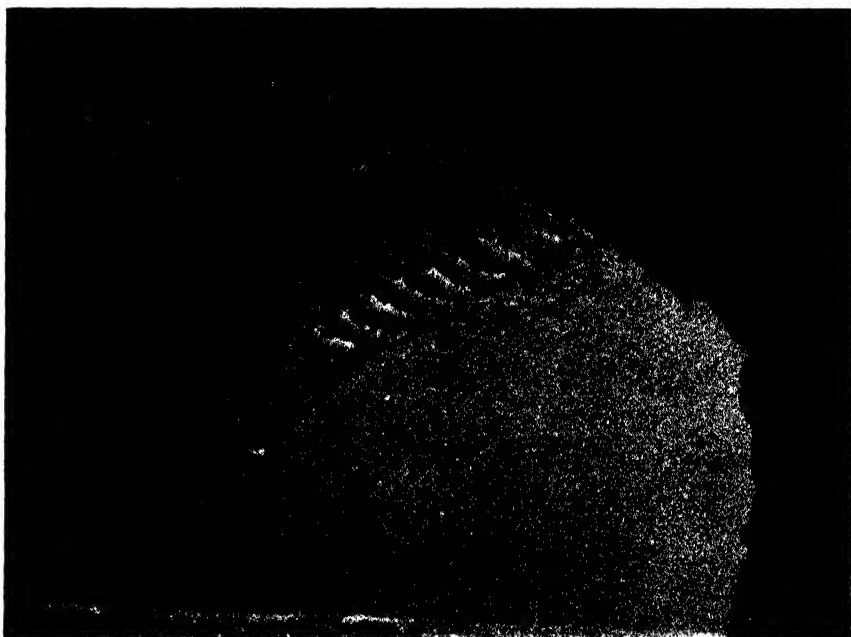
*Fig. 146. Open-hearth furnace roof showing spalled brick held in position.*



*Fig. 148. Section through open-hearth furnace roof showing guttering above back wall.*



*Fig. 149. Alumina probes after simultaneous insertion through two points in an open-hearth furnace roof, showing marked difference in iron oxide pick-up.*



*Fig. 151. Split roof on 80-ton open-hearth furnace after 7 weeks (98 casts) showing superiority of low alumina silica brick—right half.*





*Fig. 152. Panels of low alumina silica brick over the taphole in a roof otherwise built of normal quality.*

Certainly a new theory is required to explain practical experience, for if it were merely a question of temperature, then fuming would not be worst at a temperature well below that reached in the later stages of the heat.

### *Combustion control*

There is no doubt that with the present rates at which open-hearth furnaces are driven good instrumentation and control is essential to economic operation. Although the measurement of fuel rate, atomising medium rate and air for combustion must be considered as primary, other measurements, notably roof temperature and furnace pressure, are also of value in maintaining roof life. A first essential is clearly to burn the gases before they escape from the furnace, and preferably before they reach the roof, but even given these conditions fuel rates must be adjusted to avoid local overheating. In general the roof pyrometer is sighted on the central region of the roof, although it is known that other parts may be somewhat hotter. Experience shows that good lives are obtained when this region is maintained at 1650°C. maximum, but roof temperature distribution studies show that the guttering region over the back wall may well be nearer 1680°C., as may also be the exit end of the roof during the latter stages of refining. Furthermore, where a vigorous boil occurs, the gases evolved are not completely burned, and may well result in both excessive temperatures and reducing conditions at the exit end. Furnace pressures have quite rightly tended to rise during recent years, due to the realisation that too low a pressure means excessive air infiltration and, therefore, fuel wastage. It should be noted, however, that wear is likely to be greater when the furnace pressure is higher, since both heat and fluxes are carried through the joints between the bricks. In order to achieve the usual ideal of zero pressure at sill level, the corrected furnace pressure at the roof centre should be roughly  $0.01 \times H$  in. w.g., where  $H$  is the height in feet of the roof centre above the sill.

The biggest single factor leading to improved roof life during recent years, has been the roof pyrometer, which if correctly designed, installed and maintained, is a reasonably reliable instrument. It normally takes the form of a thermopile or photocell housed in a suitably cooled casing, and focussed *via* a lens on a particular area of the roof. The millivoltage arising from the impact of the radiation is converted to a temperature scale on a recording instrument. The choice and calibration of instruments is again a specialised subject, but the general procedure is quite simple, the instrument as purchased being checked by means of a disappearing filament type pyrometer, which in turn is

calibrated against a standard tungsten filament lamp. Experiments, in which the readings given by such an instrument throughout a cast were compared with those obtained by a carefully calibrated Pt.-Pt. Rh. thermocouple, introduced through the same area of the roof, showed a remarkable degree of agreement (fig. 153). Although there was appreciable scatter, particularly during the charging and melting period, the

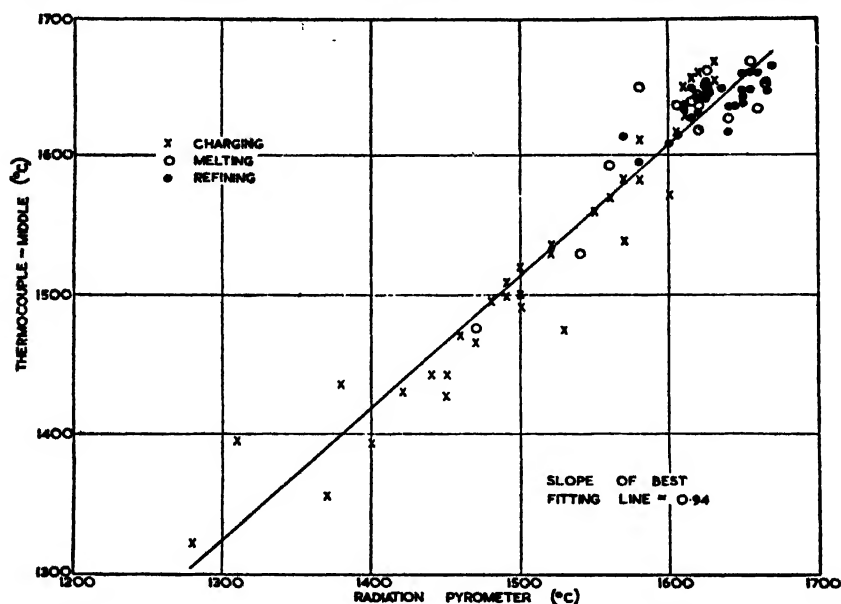


Fig. 153. Correlation between radiation pyrometer sighted through back wall and thermocouple inserted through roof.

average difference between the thermocouple and roof pyrometer readings in the above 1600°C. region, was only 8°C. Greater accuracy would scarcely seem necessary in view of the fact that other parts of the roof may be at 30°C. or 40°C. higher temperature.

The history of roof pyrometry is one of numerous teething troubles, e.g., dirty lenses and overheating of detecting elements. Those who insist that roof pyrometry is either impracticable or unreliable are generally those who have failed to overcome such installation or maintenance problems. Given a satisfactory installation good results are obtained, provided the instrument is checked at least once a day, and preferably once a shift. Unless such maintenance is intended, the roof pyrometer had far better not be installed, since it will not only be

misleading but positively dangerous: if for any reason it reads low, the melter by following it may damage his roof, whilst if it reads high he will run his furnace at an unnecessarily slow rate.

### *Metallurgical load*

Since the function of the open-hearth furnace is to convert pig iron and scrap into high quality steel, the amount of work to be done will clearly depend on the impurities present in the materials charged. The main constituents to be removed are carbon and silicon, and the time required depends not only on the amount in which they are initially present, but also on the carbon required in the final bath and the amount of sulphur and phosphorus present. The amounts of these latter depend both on the percentage of pig in the charge and on the phosphorus and sulphur contents of the pig iron, and consequently vary considerably from one plant to another. British raw materials tend to be very high in these constituents and demand an increased amount of lime to remove them from the metal. Thus furnaces frequently operate in the United States with a slag depth of only 2 to 3 in., whereas their British counterparts may be carrying as much as 6 to 7 in. If, as has been suggested, there is a drop in temperature through a basic slag of the order of  $10^{\circ}\text{C.}$  per in., then for the same metal temperature, the top surface of the slag and, therefore, that of the roof, is likely to be as much as  $30^{\circ}\text{C.}$  higher—a very substantial difference in view of the closeness of any open-hearth furnace roof to the melting point of silica.

Roof temperature charts show that whilst charging is in progress, really high roof temperatures are rarely obtained, but that when the refining stage is reached, fuel has normally to be cut back in order to avoid overheating of the roof. If this soaking stage, when the roof is working at its maximum safe temperature, is a long one, as for example in the manufacture of low carbon (dead-soft) steels, then the wear on the roof is likely to increase even more than would be expected from the extra time involved in making these special qualities. If oxygen injection is employed to speed up carbon removal the refining time is reduced but roof life may be further decreased by excessive fuming and the spitting of slag from the bath on to the roof.

### *Normal causes of failure*

It is generally accepted that provided excessive temperatures are not carried, the wearing of a silica roof occurs due to gradual solution of the surface layers by the iron-oxide picked up, the reaction product

dripping away as it forms. During the first weeks of operation, the roof undergoes certain changes, sometimes referred to as maturing or seasoning. What was not realised until relatively recently, is that the cone melting point of the working face may actually rise above that of the original brick in spite of the absorption of iron-oxide which occurs. This is a possible explanation of the melter's observation, that difficult charges should not be made on a brand new roof.

A great deal has been published (*see* bibliography) on the changes which occur in roof bricks and which, as will be seen from fig. 154, p. 357, are quite obvious even to the naked eye. Bricks develop a zoned structure, the zoning becoming clearer with longer use. The actual thickness of the zones varies with the residual thickness of the roof, but it is roughly true that the zones continue to be present even in quite small remaining thicknesses, the different colours (varying from grey at the working face through black to yellow and reddish yellow at the cool end), maintaining a proportionality. As many as 9 different zones have been specified, *e.g.*, by Harvey, but for all practical purposes, the used brick may be broken down into four main zones, as follows:

- A. The grey working face.
- B. The black section between A and the so-called transition zone C.
- C. The relatively narrow pale yellow zone between B and the cool end of the brick.
- D. The reddish section at the cool end.

In the present studies, which were carried out on a roof brick from a 300-ton tilting furnace, the B zone was sub-divided into 2 sections, B and B<sub>2</sub>, as other properties suggested differences between the two parts of this zone. The properties of this brick, including chemical analysis, are set out in Table XXVIII. The changes observed due to service may be summarised as follows:

- (1) That part of the working face which is above 1470°C. (the cristobalite-tridymite transition) changes to a network of cristobalite crystals. Although considerably higher in fluxes (notably iron oxide and lime) than the original brick, the working face is more refractory and apart from occasional large holes more impervious than the original brick. Furthermore, it normally has a glazed surface.
- (2) Big differences are found between the thermal expansion characteristics of zones A and B and zones B<sub>2</sub> and C and it is not surprising that if the old roof is heated up too quickly it is likely to spall at one or other of these interfaces.

TABLE XXVIII  
PROPERTIES OF ZONES IN USED SILICA ROOF BRICKS

	Zone A	Zone B	Zone B <sub>2</sub>	Zone C	Zone D
Length of the zone (cm.) ..	3.0	4.0	6.5	1.5	8.0
Apparent porosity (per cent.) ..	17.9	16.3	7.5	23.5	23.7
Bulk density (g.p.ml.) ..	2.09	2.04	2.10	1.81	1.81
Apparent solid density (g.p.ml.) ..	2.55	2.44	2.28	2.37	2.37
Permeability to air (perpendicular to zone interface and through the whole of the zone (c.g.s. units) ..	0.431*	0.131	0.004	0.056	0.046
Refractoriness:					
(a) Cut cone ..	1730°C.	1710°C.	1700°C.	1690°C.	1700°C.
(b) Crushed cone ..	1700°C.	1690°C.	1690°C.	1686°C.	1690°C.
Microscopic examination ..	Cristobalite, magnetite, glass	Tridymite, magnetite, glass	Tridymite, quartz, glass. Unidentified mineral of high birefringence	Similar to D	Essentially similar to unused brick (25 per cent. raw quartz)

\* As low as 0.0002 in some samples.

- (3) More detailed studies show that there is a tendency for fluxes to concentrate in a narrow zone at the interface between B<sub>2</sub> and C. This explains why if the zone becomes exposed, *e.g.*, due to localised spalling, a sideways attack may occur, which results in a layer of the roof falling away. A dramatic example of this is shown in fig. 155, p. 357.
- (4) The crystal size of the cristobalite in zone A and tridymite in zone B, as revealed by the microscope (figs. 156 and 157, p. 358) and X-ray examination (figs. 158, and 159, p. 359), is very high compared with that of the unused brick (fig. 160, p. 359).

Studies carried out on silcrete bricks show similar changes, but indicate that although alumina migration again occurs the amount even in the low melting point zone is still below that present even in an unused brick of normal type. This observation, which was confirmed by melting point determinations on cones cut from the zones, suggests that sideways attack *via* the flux zone is much less likely to occur with a super-duty roof than with one of more normal quality.

#### *Abnormal causes of failure*

At one time a substantial proportion of roofs were damaged by improper heating up. Practice varied between those who took no precautions whatever, even using wood fires (including railway sleepers) to warm up a new furnace, and those who took an unnecessarily long time and, therefore, lost production. During recent years many firms have standardised their procedure, *e.g.*, the schedule suggested by Dodd, which was particularly designed to take care of the alpha-beta cristobalite change, has been widely adopted. This schedule which enables the furnace to be gassed in approximately 24 hours, is as follows:

From 20° to 200°C.	..	Rate of heating = 25°C. per hour
„ 200° to 300°C.	.. „ „ „	= 12°C. per hour.
Above 300°C.	.. „ „ „	= 40° to 50°C. per hour

Those who have followed this schedule conscientiously do not appear to have experienced spalling trouble, but certain plants, nevertheless, still use much longer periods, *e.g.*, 48 to 72 hours, with a new roof. More recently American workers have gone in for far faster heating up, even tapping a first cast from a furnace within 24 hours of starting from cold.

The temperature control is generally carried out by connecting a thermocouple pushed through a hole in the roof to a recorder, and arranging for the operator to control the heating so as to follow a line

drawn on a chart. Even this instruction is not apparently always adequate, since operators who have conscientiously followed the heating rate have not been concerned when for some reason, say doors or chimney damper being opened, a sudden fall below  $300^{\circ}\text{C}.$  occurs. It should, therefore, be stressed that whether the furnace is being heated up or being cooled through the critical  $200^{\circ}$  to  $300^{\circ}\text{C}.$  during which cristobalite changes its form, the rate involved must be low. At one time coal fires, and the above mentioned wood fires, were employed, but today most furnaces are heated up using a series of perforated pipes passed through the doors, through which coke-oven gas is led to form small jets in the furnace.

Even a matured roof is in no way proof against spalling, particularly in view of the critical differences observed between the zones and, therefore, old roofs when allowed to cool should, strictly speaking, be brought up on an equally tight schedule. Frequently no such precaution is taken and no calamitous damage done, presumably because the roof being glazed over remains in position even though possibly badly cracked at some distance behind the working face.

Where a roof or side wall has to be patched with silica bricks while the furnace is still in operation, the amount of spalling which occurs can be greatly reduced by boiling the bricks in tar (or dipping them in creosote) prior to use. The advantage gained can scarcely be due to the bonding effect of the tar-coke, since no such coke would be formed with creosote. It is probably the result of a reduced rate of heating through the cristobalite range, due to the heat required to evaporate the impregnating liquid, whose boiling range mainly falls within the critical expansion range  $200^{\circ}$  to  $300^{\circ}\text{C}.$

Mention should also be made of the excessive damage that can be done in a very short time due to localised impact of flame on the roof. Fig. 161, p. 360, shows, for example, the 'silicicles' formed on a roof due to such overheating. If not observed shortly after it starts the melting process appears self-accelerating and 'silicicles' reaching almost from the roof to the bath may be produced. In general such damage is the result of flame deflection from an unfortunately placed scrap pile, but it may also occur due to the fuel rate being excessive.

#### (d) LINES OF IMPROVEMENT

The following are some of the ways in which roof life might be increased without loss of production:

- (1) The use of silica bricks of even higher bulk density—lower porosity.



- (2) The use of bricks made from quartzites containing even less fluxes, in particular alumina and alkalis.
- (3) The use of basic refractories as in all basic furnace construction (*see end of chapter*).
- (4) The extended use of roof pyrometry and automatic control, with particular reference to the choice of the control point or points.
- (5) The re-design of furnaces to give improved flow pattern and in particular to extend the part of the roof (at present approximately one-third) protected from flux deposition by the incoming air stream. Additional protection may be obtainable by the use of "moving curtains" of air on the inside of the furnace (*see for example Brit. Pat. 704,321*).
- (6) Control both of combustion and metallurgical operation to minimise iron-oxide emission from the charge.

## THE BACK WALL

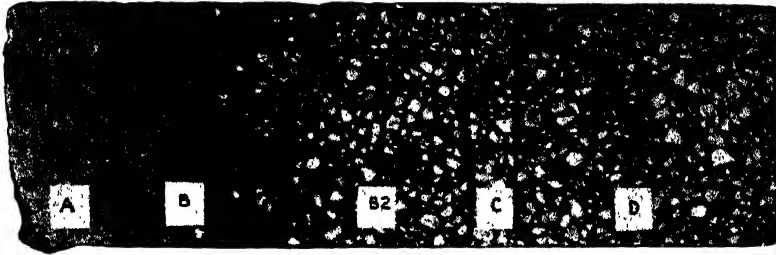
(Section 12)

### (a) CONSTRUCTION

Relatively few vertical back walls are now built, but on the other hand many furnaces once fitted with fully sloping (Naismith) back walls are now only semi-sloping, due to the encroachment of the hearth on the back wall demanded by increased capacity. In the original Naismith patent, the wall is only slightly steeper than the expected angle of rest of dolomite and can, therefore, be fettled to the top. With semi-sloping walls only the lower part can be protected, the top half being built vertical.

Numerous methods of construction are employed for vertical and semi-sloping walls, a common one being the use of 14 in.  $\times$  4½ in.  $\times$  3 in. header bricks. Bonded walls of 2½ in. or 3 in. squares with header and stretcher courses are, however, also popular. The lower half of the wall is generally made thicker than the top half, say 18 in. instead of 14 in. in order to get mechanical stability and reduce the risks from undercutting at slag level.

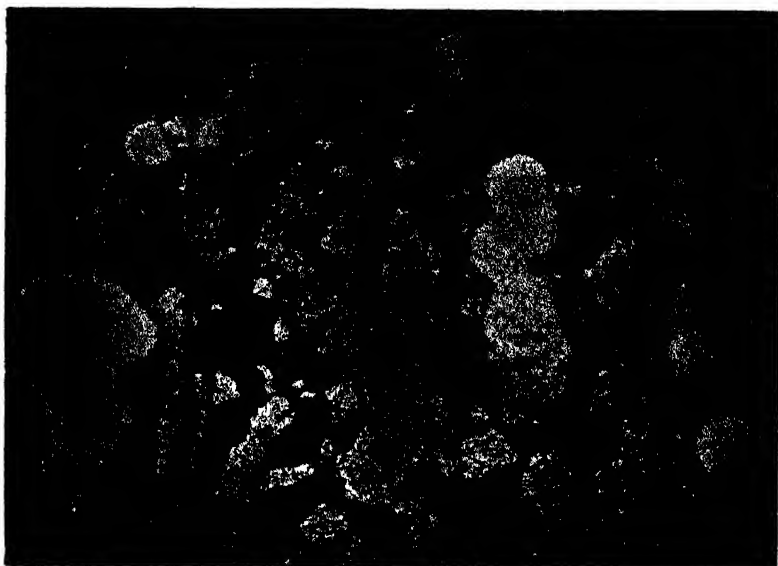
Numerous methods of tying a vertical section to the girder structure to increase stability have been developed, *e.g.*, the use of steel strips, as shown in fig. 162. It will be seen that these are inserted at intervals between the bricks and are then twisted over rods set in slots in the vertical buckstaves. Stability can be further increased by building walls on the "batter", *i.e.*, with the bricks set at a slight angle to the horizontal, so that the wall tends to fall outwards rather than inwards.



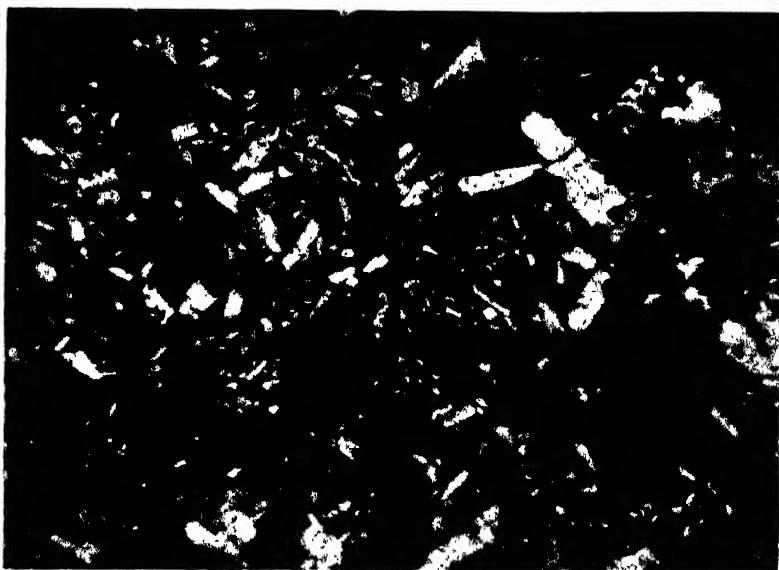
*Fig. 154. Zoned structure of silica roof brick after use in an open-hearth furnace. Zone A: cristobalite; zones B and B2: mainly tridymite; zones C and D: similar silica modifications to unused brick.*



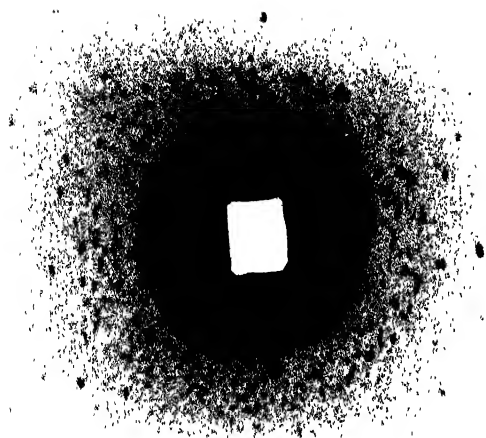
*Fig. 155. Sideways attack on silica roof bricks leading to separation of working face.*



*Fig. 156. Cristobalite network in zone A of a silica roof brick after fifteen weeks' service on a basic open-hearth furnace. Ordinary light  $\times 40$ .*

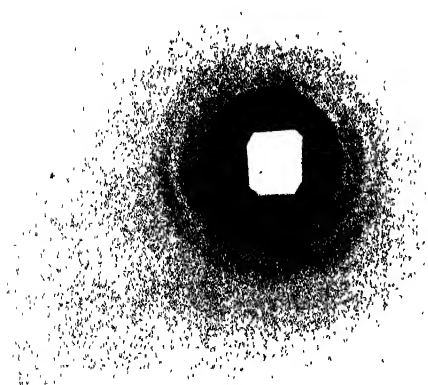
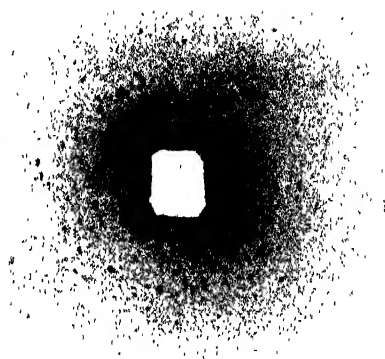


*Fig. 157. Tridymite in zone B of a silica roof brick after thirty weeks' service on a basic open-hearth furnace. Ordinary light  $\times 40$ .*

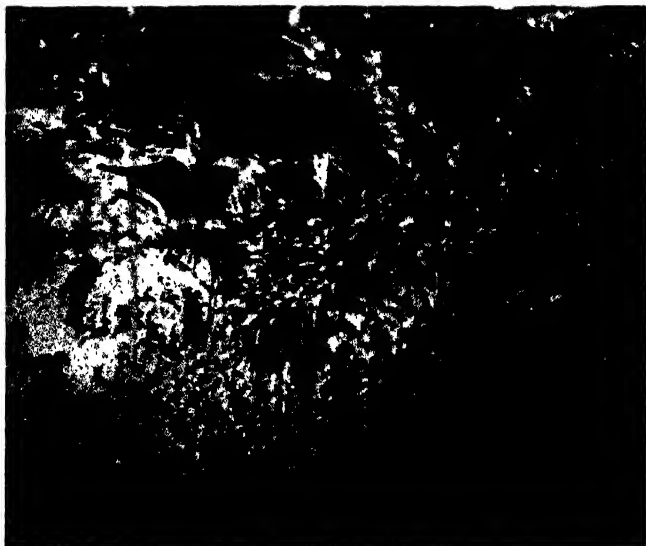


*Fig. 158. Monochromatic X-ray pinhole photograph of zone A of a used roof brick showing coarse crystallinity of cristobalite.*

*Fig. 159. Monochromatic X-ray pinhole photograph of zone B of a used roof brick showing coarse crystallinity of tridymite.*



*Fig. 160. Monochromatic X-ray pinhole photograph of unused silica roof brick. Smooth rings indicate fine crystals.*



*Fig. 161. "Silicicles" formed on a basic open-hearth furnace roof due to local overheating.*

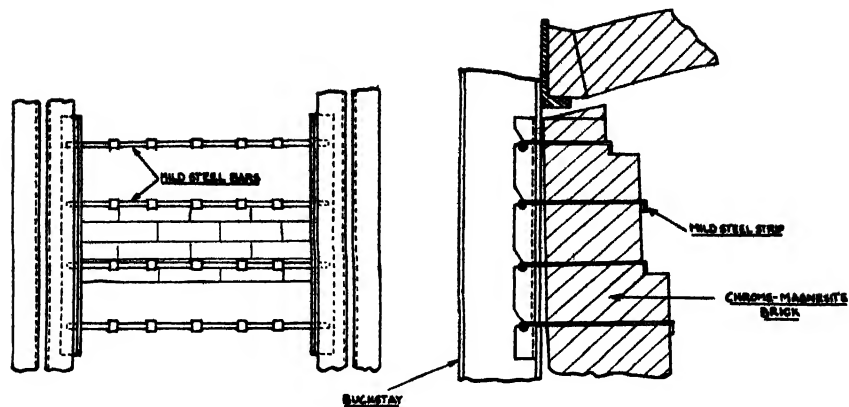


Fig. 162. Chrome-magnesite back walls tied to buckstaves by mild steel strips and rods.

Thermal expansion is generally taken care of by means of 2 or more wide vertical joints, the total allowance being approximately 1 per cent. or  $\frac{1}{8}$  in. per foot of wall, *i.e.*, roughly the thermal expansion of basic bricks up to  $1000^{\circ}\text{C}$ .

#### (b) MATERIALS

At one time silica bricks were frequently employed for back wall construction, but with faster driving their life became absurdly short. Virtually all basic furnaces are now built with basic back walls. In Great Britain, chrome-magnesite bricks are still favoured, though metal-cased magnesite tubes have given even better results under particularly severe conditions. Substantial use is also made, particularly in the United States, of unfired chrome-magnesite and magnesite-chrome bricks, generally of the metal-clad type.

Many other materials have been tried, *e.g.*, forsterite and semi-stable dolomite, but their life is generally appreciably shorter than that obtained with chrome-magnesite bricks, and their continued use has, therefore, not been found economic.

With fully sloping back walls, the working face usually consists of dolomite or magnesite fettling material, whilst the brickwork close to the case, particularly in the lower regions, is normally fireclay. In between a variety of basic bricks, *e.g.*, magnesite or chrome-magnesite, can be employed.

Back walls in tilting furnaces are also built of magnesite or chrome-magnesite bricks, but are built up at frequent intervals by throwing

chrome-magnesite or other basic cement through the doors on to the back wall when the furnace is tilted. The conditions in these furnaces differ markedly from those in fixed furnaces, in that during the tilting operation the slag rises to substantial heights over the back wall.

The comparative properties of different types of bricks used in back walls and also in front walls and ports are set out in Table XXIX.

#### (c) LIFE AND CAUSES OF FAILURE

A typical life for a semi-sloping chrome-magnesite back wall in a fixed furnace is about 13 weeks. This is roughly the life of the roof, and consequently the two are normally replaced together at an intermediate repair. On tilting furnaces, where extensive fettling with cement occurs, lives of twice this length are not uncommon, whilst with full sloping back walls even a year is not exceptional. During these periods, however, local repairs may be carried out on the hot furnace, *e.g.*, the top few courses of the back wall may be replaced with new bricks.

Analyses of used bricks removed from back walls generally show a marked pick-up of iron oxide and lime and a relatively small increase in silica. Indeed certain chrome-magnesite bricks actually show less silica in the working face than was present in the original material. Failure is mainly due to spalling and with chrome-magnesite bricks part of this would appear to be due to the bursting associated with solid solution of iron oxide in the chrome grains. When the furnace is cooled, substantial areas of the wall may shell away. At the top of the back wall there is often also substantial attack due to silica drip from the roof, which is not surprising in view of the fact that the worst roof wear occurs in the region immediately above the back wall. This complication is one of the major difficulties in using materials such as dolomite that are rapidly fluxed by highly siliceous slags. The wear is generally worst however at the slag level, particularly if the banks are insufficiently fettled, and may even lead to complete collapse of the wall above. In some plants the toe of the skewback is protected by using chrome cement between the basic brick and the silica roof. Straight chrome bricks, which were originally developed as a neutral course between magnesite in the hearth and silica walls above are no longer required, since the chrome-magnesite brick itself is essentially neutral.

#### (d) LINES OF IMPROVEMENT

- (1). The extended use of metal-cased unfired bricks tied to the girder structure by welding.
- (2). The use of improved chrome-magnesite or magnesite bricks of a type at present reserved for use in the roofs of all-basic furnaces.

TABLE XXIX  
PROPERTIES OF BASIC OPEN-HEARTH FURNACE BACK WALL, FRONT WALL AND PORT BRICKS

	Chrome-magnesite		Silica		Forsterite		Special magnesite	
	M.C.17		S.16		X.8		M.1	
Code No.								
Apparent porosity, per cent. . .	26.6		21.6		20.3		22.2	
Bulk density:								
g.p.ml. . . . .	2.74		1.89		2.76		2.76	
lb. per cu. ft. . . . .	171		118		173		173	
Apparent solid density g.p.ml. . .	3.73		2.33		3.46		3.54	
Cold crushing strength, on-end, lb. per sq. in. . . . .	1990		6350		3170		1470	
Permeability to air, perp. 9 in. × 3 in. face (through 1 skin), c.g.s. units . . . . .	0.036		0.043		0.037		0.088	
Permanent linear change on reheating, 2 hours at 1500°C. . .	0.7% (exp.)		0.1% (exp.)		0.4% (exp.)		0.25% (con.)	
Refractoriness-under-load—1600°C.—25 lb. per sq. in.—1 hour . . . . .	5.7% deformation		0.3% deformation		6.2% deformation		1480° 1630° 1630° } (50 lb. per sq. in.—rising temperature)	
Thermal shock resistance (900°C.) test . . . . .	30 +		1 (450°C. test)		7		30 +	
Notes	Bursting expansion substantial		1710° melting point. 5% raw-quartz		Resistance to iron oxide greater than silica, but less than magnesite or chromemagnesite		Grecian magnesite base	
Position of use	Front walls Back walls Ports		Front walls Back walls Ports		Back walls		Ports	



## THE FRONT WALL

*(Sections 13, 14, 15 and 16)*

### (a) CONSTRUCTION

The front wall normally consists of a series of brick pillars on the charging side of the furnace, together with door frames and doors, the latter generally numbering 3 on a furnace of 100-ton capacity or less, and 5 on larger furnaces. In addition there may be wickets or small doors for inspection and cleaning of the port slopes. The general method of building is similar to that employed in the vertical back wall, but the shortness of the sections and the impact of the material charged into the furnace make a satisfactory construction considerably more difficult. Charging pans are frequently piled up with bulky and very springy scrap, which can do damage to the front wall even if the entrance is protected by means of a water-cooled frame. The front wall, like the back wall, is generally at least 14 in. to 18 in. thick, and is often tied in to girder work, for example by means of metal strips. Stability of the individual pillars is enhanced by building them thicker and broader at the bottom so as to get a "batter" not only on the inside but also in the door jambs. The door arches are generally built of silica and can be strengthened by the use of tongued and grooved blocks. Shut-downs due to arch failures can be, of course, completely eliminated by resort to water-cooled lintels, but it should be noted that this procedure makes the problem of constructing a stable intermediate pillar all the more difficult. Sloping front walls, similar in principle to the Naismith sloping back wall, have been suggested, but their application to existing furnaces is normally ruled out by the alternative evils of reduced bath capacity or protrusion over an already congested stage. At least one firm has tried out the complete replacement of the front wall by a series of sliding doors. Such an arrangement should permit of the charging of particularly large scrap and would eliminate the problem of front wall pillar stability. It would, however, be expected to lead to substantial increases in water-cooling and consequently in heat losses. With most modern furnaces the door frames and sometimes the doors themselves are water-cooled. This latter adds substantially to door life and to the comfort of the operator, but again adds to other costs. All such changes must be budgetted in terms of overall costs, bearing in mind that whereas water-cooling normally means an increased loss of thermal units as hot water, it may actually lead to a fuel saving if it permits of harder driving or decreases the time during which the furnace is off for repairs. A further advantage is gained, in that the good seal of the water-cooled door and frame enables a higher furnace pressure

to be carried and, therefore, minimises air infiltration and consequent fuel wastage. A better solution may well prove to be the use of so-called steam cooling as developed in Germany. Here the water cooling elements are an integral part of a boiler system and the heat picked up is therefore partially recovered.

#### (b) MATERIALS

The materials used in the pillars are generally similar to those employed in back walls, though some plants still employ silica for the above sill sections. The position of the front wall in relation to the stage permits of hot repairs being carried out, including parging of the jambs. With large tilting furnaces this is often a vital part of front wall maintenance, as much as a ton of chrome-magnesite cement being used per cast made. The cement is made by grinding a chrome-magnesite mixture with water until a fine and sticky condition is obtained. The job of parging is extremely arduous and it is doubtful whether more than half the material applied stays in position for more than a few minutes. Even so it enables silica bricks to survive far longer than would otherwise be possible.

A useful and cheaper alternative cement was developed during the Second World War, by mixing together 80 parts of serpentine with 20 parts of magnesite. After fine grinding in a pan mill and adding water to the extent 20 per cent. such batches have a very sticky consistency and adhere quite well even to a hot wall. Incidentally it is essential that the magnesite fraction should be fine, so that it reacts readily with the serpentine to yield forsterite. Where pan milling is practised the magnesite should therefore be added in an already fairly finely ground state.

#### *Doors*

Door bricks offer an interesting example of the way in which the survival of the fittest has resulted in bricks with certain special properties being selected for use under particular conditions. The properties of a number of such door bricks are given in Table XXX, from which it will be seen that all of them have a particularly high thermal shock resistance. That bricks having these properties should survive is not surprising in view of the fact that cold doors are frequently fitted on furnaces at temperature of 1600°C. upwards. Where a furnace is warmed up from cold even silica bricks are satisfactory, although they would be useless for hot application. More recently considerable development has taken place in rammed doors, the key idea being the

TABLE XXX  
PROPERTIES OF FIRECLAY BRICKS USED IN OPEN-HEARTH FURNACE DOORS

<i>Code No.</i>	<i>F.1</i>	<i>F.2</i>	<i>F.5</i>	<i>F.27</i>
Apparent porosity, per cent. . . . .	25.9	21.7	23.0	18.9
Bulk density:				
g.p.ml. . . . .	1.94	1.98	1.98	2.05
lb. per cu. ft. . . . .	121	123	123	128
Cold crushing strength-on-end, lb.				
per sq. in. . . . .	3130	4140	4110	1890
Permeability to air, perp. 9 in. $\times$ 3 in. face (through 1 skin), c.g.s. units . .	0.46	0.013	0.009	0.40
Permanent linear change on reheat- ing, 2 hours at 1410°C. . . . .	-0.6% (con.)	-0.6% (con.)	4.8% (exp.)	1.1% (exp.)
Refractoriness-under-load, 50 lb. per sq. in.:				
Initial softening . . . . .	1360	1220	1140	1270
Rapid softening . . . . .	1490	1450	1260	1410
Fail temperature, °C. . . . .	1570	1540	1440	1600
Thermal shock resistance (900°C. test) . . . . .	30+ Over	30+	30+	30+
Refractoriness . . . . .	1730°C.	1680°C.	1680°C.	1760°C.

use of a water-cooled door frame, to which steel studs have been previously welded or screwed. Basic or neutral cement, *e.g.*, chrome plastic, is then rammed in layers between and over the studs. Such doors give lives far in excess of those made from fireclay refractories but also cost a great deal more. According to certain plants, their use is definitely economic, though as yet quite limited.

#### (c) LIFE AND CAUSES OF FAILURE

The pillars in a front wall may last a whole campaign of, say, 13 weeks, but the door jambs and arches may well have to be replaced after only 6 weeks. Such repairs usually have to be done while the furnace is in operation, or at least extremely hot, and even with the great skill shown by "hot hands" are unlikely to last as well as the original bricks. An analysis of stoppages on open-hearth furnaces frequently reveals several such repairs towards the end of the campaign and anything that can be done to increase front wall life, or improve repair techniques, would undoubtedly pay dividends. Where silica bricks are employed, spalling can be considerably reduced by prior boiling in tar or even dipping in pitch-creosote. The heat required to boil off these additions markedly reduces the rate of rise of temperature and thus the cracking risk. The use of fireclay bricks for such hot repairs is to be deprecated, since even an odd brick falling on to the dolomite hearth may be sufficient to cut a hole through it and allow the contents of the furnace to break out. Door life is extremely variable, being as low as 80 hours on some furnaces, but as much as 3 weeks where the doors are water cooled. Analysis of the working face of used door bricks suggests that, as in other parts of the furnace, corrosion is mainly due to iron oxide and lime, which are presumably picked up from the bath by the relatively high-speed flame and reach the doors themselves through the subsidiary eddies formed between the door jambs.

#### (d) LINES OF IMPROVEMENT

Numerous attempts have been made to improve the front wall apart from the radical one of abolishing it altogether and replacing it by a series of doors. The greatest hope would seem to be some form of interlocked panel, that would not only be structurally independent of the bonding action of cement, but might even enable prefabricated panels to be installed as hot repairs. As with the back wall, the greatest hope from the refractory angle lies in the use of improved type basic bricks, *e.g.*, the electro-cast type referred to in chapter V.

## GAS AND AIR ENDS

(Sections 1 and 3)

### (a) CONSTRUCTION

These portions of an open-hearth furnace are sometimes referred to as the target areas, a well earned title in view of the fact that they are perpetually bombarded by the exit gases as they turn through 90° or more on leaving the furnace chamber and enter the downtakes. Those who have inspected a furnace through the peep-holes in the ends know that some at least of the particles are a substantial size and may travel for several feet on leaving the furnace through these holes. There is little doubt that a large proportion of the coarser droplets in the gas stream are deposited on the end walls and the conditions are therefore very severe. It is for this reason that the air ends have often been used as points where tests on new materials can be quickly brought to a conclusion. Although something can be done to reduce such attack by re-design, *e.g.*, sloping ends, the effect may be to carry this wear into a lower part of the furnace, where repairs become more difficult and the net gain is, therefore, a doubtful one. In general these walls are built in silica or basic brick and are of 14 in. to 18 in. thickness.

A further problem arises with gas ends, in that they must periodically be opened up for inspection of the port slope and rabbling or fettling of it. Ideally a door over the end would be useful but the more general practice is still to use a panel of silica bricks, some of which generally fall into the slag pocket when the end is opened up.

### (b) MATERIALS

Until recently furnace ends were largely built of silica bricks. With harder driving and particularly the use of driven fuels, the wear in these regions has become so excessive that recourse must be had to basic, *e.g.*, chrome-magnesite or metal-case magnesite bricks. Numerous comparisons of the life obtained with silica and basic bricks in this position leave no doubt that this is one of the locations where a more expensive brick can pay its way. Incidentally the basic panel should be carried far enough down the uptake to ensure that no serious reaction occurs at the junction between the basic and the silica sections. In many furnaces the wear on the pit-side area is found to be far greater than on the charge-side and in such cases the pit-side is sometimes built with basic bricks, whilst the charge-side although built of silica lasts equally long.

**(c) LIFE AND CAUSES OF FAILURE**

Most of the damage to gas and air ends appears to be due to iron oxide with a lesser amount of lime. Samples taken in the uptakes show that periodically, however, other materials, *e.g.*, dolomite from fettling, or fluorspar added to liquify the slag, may also be present in substantial quantities. Zinc-oxide and lead, which are found in considerable quantities in the checkers, do not appear to remain in this region of the furnace, presumably due to their high volatility.

**(d) LINES OF IMPROVEMENT**

Since the temperatures in the ends are of the same order as those in the roof, there is a strong case for the use of top grade refractories. At present good chrome-magnesite or metal-case bricks appear best, but the conditions might well warrant the production of bricks of lower porosity even if this were obtained by the sacrifice of other properties, such as refractoriness-under-load.

**PORTS**

Section 10A	..	..	Gas port arch
„ 10B	..	..	Gas slopes
„ 10C	..	..	Wing walls
„ 10D	..	..	Air port arches (if present)

**(a) CONSTRUCTION**

The port in a producer gas or mixed gas furnace is essentially a semi-circular tunnel passing from the gas end into the laboratory of the furnace. It may be built as a series of rings or from solid blocks to form a central hole or holes. Air is introduced above the port either by using a venturi type ramp to throw it down into the gas stream, or by relying on the entraining action of the flame to draw its own air from the regions above and at the side of the port. The difficulty of maintaining brick ports has led to numerous special types being developed, *e.g.*, those having a series of 1 in. mild steel cooling pipes in the nose, and those constructed with a massive water tank with or without a brick lining. Thus the short Maerz type port is simply a water-cooled horseshoe, whilst the Blair port, which may be 10 ft. or more in length, is generally lined with at least a 4½ in. ring of bricks and is only exposed to the furnace at the horseshoe end, facing inwards. If silica bricks are used no particular trouble is experienced with mechanical stability, but the rate of wear is so great that it has become customary to build the port longer than desired and to continue using it until it is much

shorter than desirable. Since flame control and, therefore, roof wear, depend very much on port length and condition, there is everything to be said for constancy, and it is this fact that has led to the far wider use of water-cooled ports of the Blair or Maerz type. One serious limitation in the use of a small gas port to get a high momentum flame is the relatively small proportion of waste gases passing out through it at the exit end. Numerous attempts have been made to overcome this difficulty, *e.g.*, the Rose patent which employs a moveable port. Few if any of these have survived due to the mechanical problems involved.

#### (b) MATERIALS

Until recently the best results with gas ports have been obtained by the use of at least a nose of basic bricks—generally chrome-magnesite or special magnesite. Where water-cooling is employed such bricks last quite well, though collapse of the port end leaving the cooling rings bare is by no means uncommon. One difficulty with the use of basic ports is the falling of refractory debris on to the gas slope. Even with a silica port sole the removal of this material is difficult and arduous, whilst leaving it there results in poor flame direction and high pressure drop on the exit end. Where magnesite bricks are used for the port end, it is desirable to use chrome-magnesite or special chrome courses for the transition to the silica back of the port blocks. Quite recently experiments have been made with the use of electro-cast materials in this position, *e.g.*, those made from fused zirconia—alumina—silica batches. The results obtained suggest that it may well prove possible to get a longer life in this position without recourse to water cooling. The price of such bricks is far higher than that of even basic bricks, but the initial cost would be largely offset by savings in water and heat.

#### (c) LIFE AND CAUSES OF FAILURE

In general an attempt is made to build a port that will last the first half of a full campaign on the furnace—normally about 13 weeks. With silica bricks this can only be done by the use of patching techniques, *e.g.*, the so-called “Scotch block” procedure, in which a steel former is put into the port mouth and a ground mixture of old silica bricks thrown over it and sintered in position. With basic bricks and ring cooling, lives of 13 weeks are quite common, whilst with the Blair port there should be no time consuming repair required unless the metal port itself fails, in which case a new one can be installed with only a

relatively short delay. Since the conditions at the port mouth on the exit end are probably more severe than anywhere else in the basic furnace, and since the alternative to a super refractory is substantial water cooling, it may well be in this region that super-refractories first make a tonnage entry into the steelplant.

#### (d) LINES OF IMPROVEMENT

As producer gas and mixed gas furnaces strive to compete with increasingly fast oil and tar-fired furnaces, the necessity for improved port life increases. For this reason and the factors mentioned above, extensive trials of super-refractories, *e.g.*, electro-cast materials, would appear justified.

### THE ALL-BASIC FURNACE

Long before the present extensive use of basic ends on open-hearth furnaces, attempts were made, originally by the Austrians, in the early 1930's, to build the whole of the above stage section of the open-hearth furnace with basic bricks. The principal objective was to enable the furnace to be run, say, 100°C. hotter than was possible with silica, and thus obtain substantially greater outputs per square foot of hearth area. It is now fairly certain that, "other things being equal", such higher roof temperature would lead to faster working, since radiation is the main factor involved and the heat transfer between roof and bath varies as the difference between the 4th powers of the absolute temperature levels. Moreover, since the furnace undergoes standing heat losses that are not greatly affected by minor changes in internal temperature, such increased output rates would be expected to result in substantial fuel savings.

It is an extraordinary thing that although all-basic furnaces have now been built in most parts of the steelmaking world, it is only on the Continent of Europe that they have proved generally economic and no adequate explanation has yet been given as to why this is so, or why life should be so critically dependent on furnace size (*see* fig. 163). Furthermore, in spite of a quite unprecedented concentration of research effort, the precise factors responsible for the failure of basic roofs still remain something of a mystery.

Since the all-basic furnace only differs from many other modern furnaces, in that the roof is built of basic bricks, it will suffice for the present to discuss the behaviour of the main and ramp roofs of such furnaces.



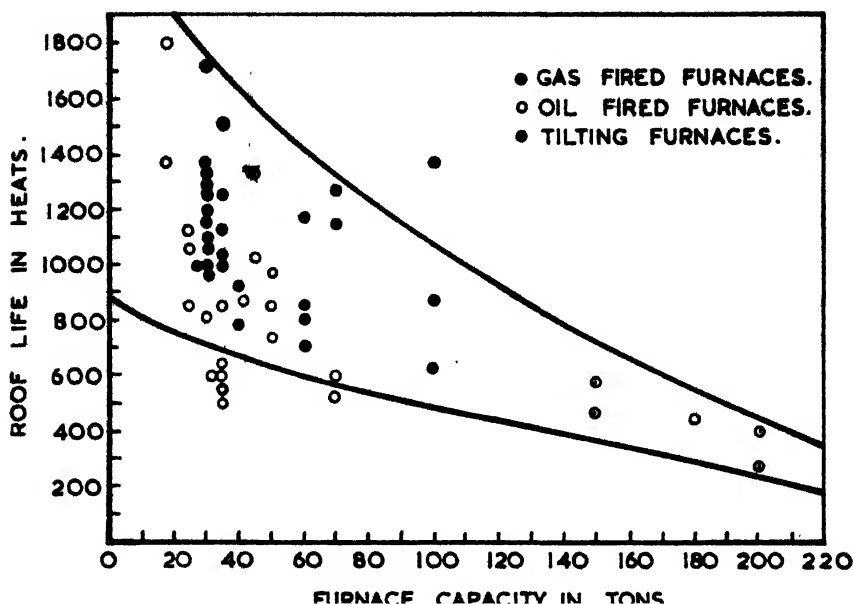


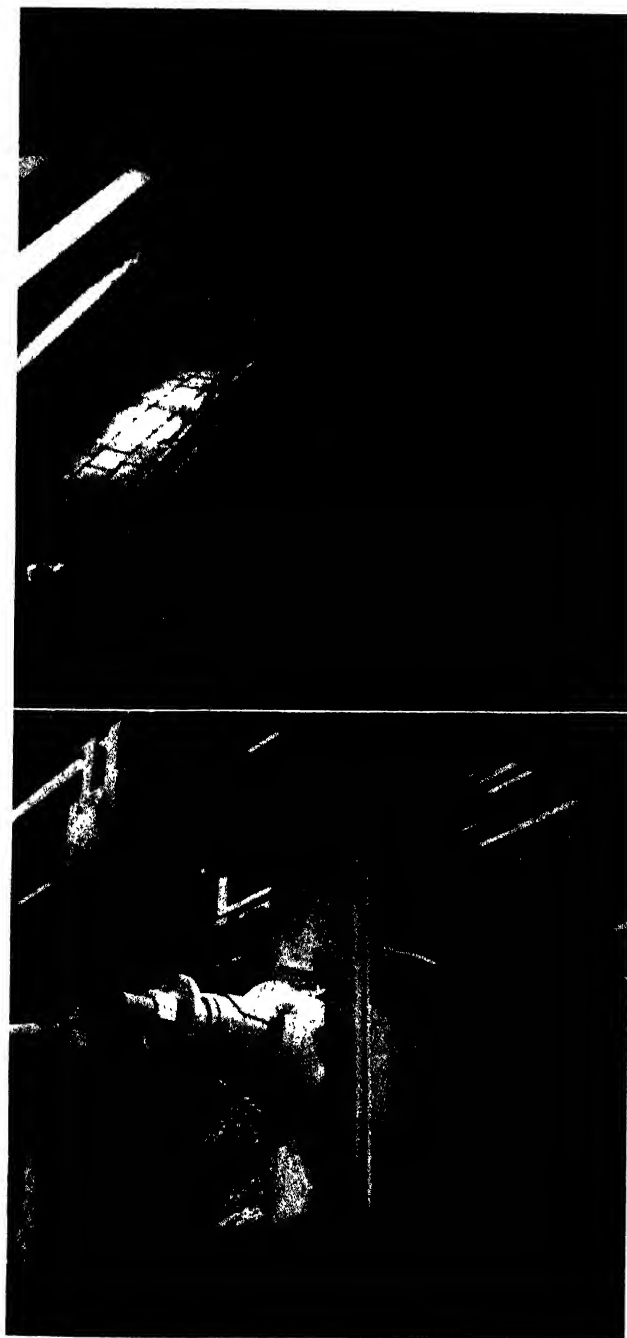
Fig. 163. Relation between all-basic furnace size and roof life in heats (after Hütter).

## THE ROOF, INCLUDING RAMPS

### (a) CONSTRUCTION

Attempts to build basic roofs in the manner adopted with silica roofs, *viz.*, simple sprung arch techniques, generally led to premature failure. As a result at least two main designs intended to ease the mechanical stresses on the brick have been developed. The first of these is illustrated in fig. 164, p. 373, which shows that with the Austrian, or so-called Continental roof, a rib construction is employed and T irons placed over the ribs are connected to cross-ties on the furnace. In the original build the roof is keyed up in the normal way on centres and the helical springs on the back wall of the furnace skewback channels are then tightened up until the roof becomes fully sprung. The principal objective of this design is the maintenance of good shape in the roof and the avoidance of large nipping stresses such as might occur where rigid skewback construction is employed.

In the American (Detrick) design (fig. 165, p. 374) no ribs are employed but each pair of bricks is hung on a heat resisting cast-iron hammer head. In this way the loading at the centre of the roof is reduced to zero (compared with about 25 lb. per sq. in. in the Austrian



(b)

(a)

Fig. 164. Continental type roof construction for all-basic furnaces (Courtesy, Austro-American Magnesite Co.).

(a). View of roof from above showing holding-down device and suspension wires.

(b). View of back wall showing screw-back channel with helical spring attachments.



*Fig. 165. American type roof construction for all-basic furnaces  
(courtesy Guest Keen Iron and Steel Co. and M. H. Detrick).*



*Fig. 166. Excessive shelling shown by all-basic furnace roof after 97 heats.*



*Fig. 167. All-basic furnace roof after repair.*

construction), whilst that at the skewbacks falls to a matter of 5 lb. per sq. in. compared with, say, 25 lb. in the Austrian design. In both types of construction steelplates or mesh are used between the bricks or steel-plated unfired shapes employed. No cement is used but on heating up the plates oxidise and the bricks are welded together, though not as effectively as with unplated silica bricks.

#### (b) MATERIALS

Numerous different types of brick have been tried out in all-basic furnace roofs, *e.g.*, chrome-magnesite, magnesite-chrome (*see* Table XIX, Chapter V), straight magnesite, magnesite with alumina additions, straight alumina and mullite, but of these only two types, *viz.*, chrome-magnesite and magnesite-chrome, have survived, the range of composition being covered between chrome (70 to 20 per cent.) and magnesite (30 to 80 per cent). Thus the original Austrian type brick was made with approximately 70 per cent. of good quality chrome ore of roughly  $\frac{1}{8}$  in. size downwards, together with 30 per cent of finely ground magnesite. Most of the American bricks contain a much lower chrome-magnesite ratio, are chemically bonded and of the steel-plated type.

Numerous attempts have been made to set-up specifications that would ensure the bricks used having adequate properties, but no satisfactory standard has as yet been produced. From time to time importance has, however, been given to the following:

##### (1) *Thermal shock resistance*

Once silica has reached red heat no further trouble is experienced due to thermal shock spalling. With basic refractories on the other hand the linear thermal expansion may lead to shock at temperatures at least up to the 1300°C. region. For this reason a minimum thermal shock resistance, *e.g.*, 30 + in the small prism spall test, has been specified by certain users.

##### (2) *Refractoriness-under-load*

The need for mechanical support largely arises from the relative weakness of basic bricks (compared with silica bricks) under load at high temperatures. It has for example been suggested that basic bricks should show a maximum of 3 per cent. contraction after 1 hour at 1600°C. under a load of 25 lb. per sq. in.

(3) *Bursting expansion*

Since part at least of the damage appears to be due to the bursting of chrome-magnesite bricks associated with solid solution of iron oxide in the chrome, bricks showing a low bursting index have been preferred.

(4) *Permanent linear change on reheating*

Bricks that are soft-fired can show appreciable shrinkage on firing say at 1700°C. A maximum value may be placed on this figure to minimise the risk of spalling due to alteration of the working face and opening up of joints consequent upon high temperature after-shrinkage.

Although it is true to say that most of the best bricks at present employed in all-basic furnace roofs would show up well in the above tests, the position is still so little understood that even better results might well be obtained from bricks having other and as yet unspecified properties.

(c) **LIFE AND CAUSES OF FAILURE**

The range of lives obtained on all-basic furnaces is truly remarkable, the number of heats varying from less than 100 to over 3000. The biggest single factor would appear to be that of furnace size, but others, e.g., the metallurgical load, are clearly of importance. What is increasingly evident is the damage done by repeated cooling down to, say 900°C., which is likely to be worse in cold-charged furnaces, particularly where rapid charging rates are employed or fuel turned off during fettling. To be economical at present price levels for basic and silica bricks and purely on a basis of refractories costs a life about  $3\frac{1}{2}$  times that of silica is required. Given this gain then the extra output and fuel saving can be considered as profit. On many furnaces, however, the lives are often little more than those given by silica roofs.

The type of wear observed varies greatly from furnace to furnace. It may be extreme, as shown in fig. 166, p. 375, where the trial of a new design led to serious nipping forces, normal, as shown in fig. 167, p. 376, or almost negligible, as observed in furnaces that run for 2000 heats or more with no obvious slabbing of the roof. Five principal theories have been advanced to explain failure, viz.:

- (1) Thermal shock due to rapid cooling.
- (2) Creep of basic bricks at temperatures of 1300°C. upwards.
- (3) Bursting due to iron oxide.

- (4) Flux concentration at a region one or two inches behind the working face leading to a localised drop in strength.
- (5) Expansion due to alternating oxidising and reducing atmospheres.

All these causes can be tackled and indeed have been, *e.g.*, by the use of coarsely graded bricks to give high thermal shock resistance, the use of low lime magnesia to give low creep values, and low flux concentration, the employment of a batch, high in forsterite or added serpentine to reduce iron oxide bursting, and of chrome ores of high stability, but none of these remedies has led to any outstanding improvement.

#### (d) LINES OF IMPROVEMENT

Although an enormous amount has been learned about all-basic furnaces, we are still very much in the dark as to the real causes of failure and, therefore, as to the correct action required. Extensive panel trials have been made in Great Britain and on the Continent of Europe, in which different bricks of known properties were compared and an attempt made to link properties with life. These are being supported by trials on large-scale roof rigs, where different designs, materials and operating conditions, can be tried out with a view to improving practice. Much could doubtless be done by reducing the amount of fluxes reaching the roof, *e.g.*, by aerodynamic design of the furnace or control of combustion and the melting process, but such changes would also improve silica roof life and, therefore, not make any marked difference to the economic ratio. Attempts to build a half-way house, *e.g.*, the Zebra roof, in which alternate rings in the silica roof are replaced by chrome-magnesite, although possibly worth while in certain plants, do not offer a real solution to the problem, if only because such a roof is still limited by the melting point of the silica section, and cannot therefore provide the real gain hoped for with the all-basic furnace, *viz.*, increased production rate.



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## Chapter IX

# THE BASIC OPEN-HEARTH FURNACE BELOW THE SILL-PLATE LEVEL

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THE CASUAL VISITOR to the steelplant who normally observes the process by looking through the furnace doors, might be surprised to know that the part of the furnace he sees is only about one-third of the total—the other two-thirds being below stage level. Even refractories technologists have tended to forget this larger part in their concentration on the maintenance of the more severely stressed furnace chamber proper. With the increasing pressure for production and the consequent rise both in fuel rates and furnace temperatures, both improved refractories and closer control of operating conditions have been necessary to maintain roof life. That this has proved practicable in many plants even with production increases of the order of 25 per cent, is due in the main to the development of improved (low alumina, low porosity) silica bricks and the introduction of reliable roof temperature and furnace pressure measurements.

The higher firing rates with their greater exit temperatures and waste gas volumes are, however, taking their toll of the downstairs section and many furnaces are now showing the urgent necessity for improved refractories in both slag pockets and checkers.

## THE HEARTH

*(Sections 7, 8 and 9)*

Great improvements have taken place during recent years in the methods of hearth installation, mainly because of a desire for:

- (1) Quicker methods of installing hearths and, therefore, greater furnace availability.
- (2) Reduced breakout risk on relatively thin hearths such as are used in Great Britain.

The former is part of a world-wide drive for increased productivity, the latter due in considerable measure to the additional stress placed on furnace hearths during the Second World War when air-raided warnings frequently demanded that metal ready for tapping be held for long periods in order to avoid glare.

## (a) CONSTRUCTION

According to Buell, hearths of up to 85 in. thickness have been employed in the United States, one such hearth consisting of 60 in. of firebricks, 12 in. of magnesite bricks, and 13 in. of ground magnesite. British furnaces tend to be at the other extreme, the brickwork in furnace bottoms often being only 9 in. thick, making with the monolith a total thickness of less than 2 ft.

Modern American practice has been summarised by Sosman, Robinson and Debenham, in the second edition of *Basic Open-Hearth Steelmaking* (A.I.M.M.E., 1951). According to these authors, insulating bricks are normally employed against the pan, and fireclay bricks laid above these and below the basic brick courses. The latter are corbelled upward at the front, back and ends of the bath, so that the overlying burned or rammed monolithic hearth refractories may be nearly uniform in depth. Expansion joints are provided between the basic bricks, which are either filled with heavy paper or covered with other bricks to prevent them being blocked by the granular bottom material. Trials have also been made of an inverted arch type construction, where shaped basic bricks are used to form the contour of the hearth, and the thickness of monolith is both small and uniform.

In British hearths insulation is also used, but is generally kept at a low level in order to avoid trouble due to the monolith itself becoming soft. The hearth bricks themselves are normally arranged (*see* fig. 168) so as to eliminate straight through joints and thus minimise the risk of a

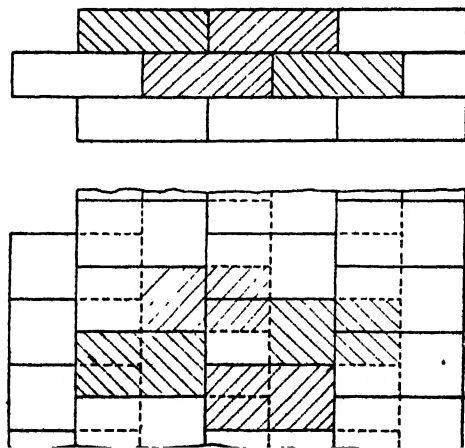


Fig. 168. Staggering of basic bricks in the sub-hearth of an open-hearth furnace to avoid straight-through joints.

breakout. As in the United States, there is an increasing tendency to build the brick sub-hearth with the surface having a contour roughly similar to that of the finished hearth and then ram or burn on an approximately even layer of monolithic material.

(b) MATERIALS

*Brick*

The properties of typical magnesite and dolomite bricks as used in furnace bottoms have already been given in Chapters III and IV. In general dense magnesite bricks, of the type first made in Austria but now quite generally available, are preferred. The porosities of such bricks should normally be less than 20 per cent. Even if this results in some loss of thermal shock resistance it is not serious, since the bricks are heated up slowly and even if cracked are unlikely to become dislodged.

Stabilised dolomite bricks made from clinker of 13 to 15 per cent. silica content have given consistently excellent results in British open-hearth furnace bottoms, and, having given equal service at a lower cost, have replaced magnesite bricks in many plants. Their resistance to hydration is at least equal to that of most magnesite bricks; a well-fired dolomite brick remaining stable even after boiling for several weeks in water.

The fireclay bricks used to cover the pan or the insulating brick layer are generally of the medium alumina type and similar in properties to those described as door bricks in the previous chapter. The insulation used varies considerably from one plant to another, but is normally of the diatomite-asbestos type. Porous silica bricks have also been employed and are sufficiently stable to be used again provided they are not subject to undue thermal shock, e.g., water quenching during removal.

*Monolithic material*

Until recently it was the custom all over the world to burn in most if not all of a basic bottom. For example magnesite hearths were installed by fettling the brick sub-hearth after heating to about 1600°C., with dead-burned magnesite grain mixed with basic slag or millscale, each layer being fully burned-in before the next was added. Similar techniques were adopted with dolomite hearths, the amount of slag or millscale being reduced in both cases as the surface layers were approached.

The factors mentioned in the introduction have led to the development of a number of alternative procedures for the installation of fully rammed hearths, which can be summarised as follows:

- (1) The so-called B.F.L.C. (Basic Furnace Linings Committee) type, in which pan-milled tar-bonded doloma is employed.
- (2) The use of dry-rammed finely graded doloma.
- (3) The use of graded magnesite together with an air-setting bond.

The original, and in some places still accepted, procedure is that in which burned dolomite or magnesite grain is thrown on to the hearth after the latter has been raised to full temperature—about  $1650^{\circ}\text{C}$ . The first layers are normally bonded either with basic slag or with millscale and each layer burned-in before the next is added. The process is a long and strenuous one, as much as a week being taken to burn-in a new bottom. Where, as in the past, hearths were employed for as long as 15 to 20 years, such a practice may have appeared justified, but the increase in fettling time and in particular in bad bottom time, of hearths of more than two years old, is now considered sufficient justification for the use of quick installation methods and frequent renewal of the monolithic section.

(a) B.F.L.C. TYPE—TARRED, PAN-MILLED DOLOMA

Co-operative research between a number of British steel companies, led to the development (*see* I.S.I. Special Report No. 33) of dolomite hearths of high packing density that gave excellent service after complete ramming, heating-up and slagging. The key idea in this

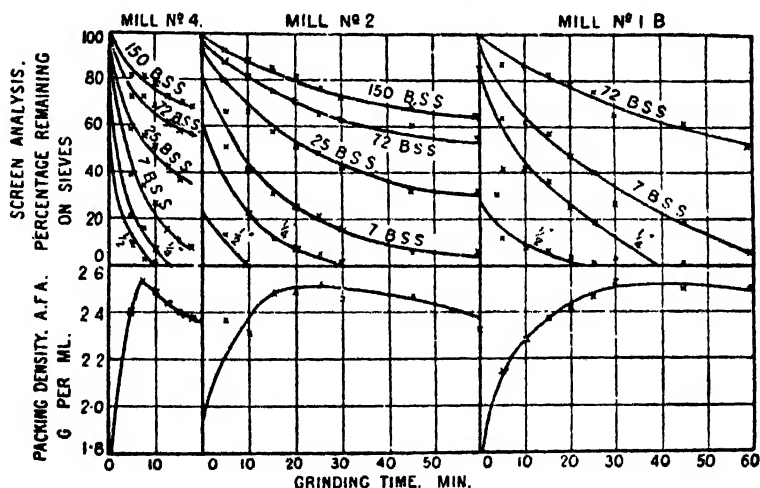


Fig. 169. Effect of grinding time on grading and packing density of doloma mixtures. Crushing load of mills shown are: No. 4—12 tons, No. 2— $5\frac{1}{2}$  tons, No. 1B—3 tons (*after* Cross, Marshall and Sarjant—I.S.I. Special Report No. 33).

TABLE XXXI  
GRINDING CHARACTERISTICS OF A SOLID-BOTTOM PAN MILL  
(No. 2, fig. 169)—5½-ton crushing load

Milling time. Min.:	0	5	10	15	20	25	30	45	60
Grading:	%	%	%	%	%	%	%	%	%
+½ in. . .	21	13	—	—	—	—	—	—	—
½ in.—¾ in. . .	41	38	22	11	7	5	1	—	—
¾ in.—7 mesh . .	19	15	21	20	18	16	15	5	5
7—25 mesh . . .	11	14	23	26	27	28	27	28	26
25—72 mesh . . .	4	8	15	18	18	16	19	27	24
72—150 mesh . .	2	4	7	9	11	11	10	9	9
—150 mesh . . .	2	8	12	16	19	24	28	31	36
Packing density (A.F.A.):									
250 g., 50 blows) g.p.ml.	1.95	2.36	2.31	2.48	2.49	2.52	2.46	2.46	2.32

particular development lay in the discovery that the packing density of crushed doloma as normally used for burning-in bottoms could be increased by 20 to 30 per cent. by controlled grinding in a solid bottom pan mill. Further that for each mill and charge there is an optimum grinding time at which the packing density reaches a maximum, further grinding resulting in no improvement, and in some cases in a deterioration. The effect of time on grading and packing density of a selected batch is illustrated in Table XXXI and fig. 169. The latter also shows the effect of mill loading on grinding rate. It will be seen that whereas with a typical 5-ton solid bottom pan mill the maximum packing density was obtained in 20 minutes, with a 12-ton mill a much shorter period—only  $7\frac{1}{2}$  minutes, was needed. Intensive research showed that the increase in packing density obtained is not purely a question of overall grading, much lower figures being obtained if the same grain size groups are made up from doloma ground in other types of mill. The high packing density is in large part due to the rounding of the particles, to give closer packing, a fact dramatically brought out by fig. 170, p. 393, which shows the result of exposing a photographic plate partially covered by  $\frac{1}{4}$  in.—7 mesh material sieved out of doloma milled for 0, 15, 30, 45 and 60 minutes respectively. The darkened areas represent the spaces between the grains, which, it will be seen, decrease very markedly as grinding proceeds. Most British steelplants now employ such material, the tar addition (5 to 10 per cent.) normally being made during the last few minutes of grinding.

The Basic Furnace Linings Committee made the following recommendations:

- (1) Well-fired doloma should be supplied to the pan mill at a size of  $-\frac{3}{4}$  in.
- (2) Before charging to the mill it is advisable to preheat the crushed material to about  $70^{\circ}\text{C}$ .
- (3) The tar should be preheated to over  $40^{\circ}\text{C}$ ., the exact temperature depending on the type of tar used. Complete finality has not yet been reached on the specification for the tar, though a relatively viscous product is favoured.
- (4) The following conditions in the pan mill should be carefully maintained:
  - (a) It should be kept as clean as possible. A run of hot ashes is stated to be satisfactory for cleaning purposes.
  - (b) The rollers should be maintained with flat faces to obtain maximum output.
  - (c) The scraper plates should be set and reset as conditions warrant.

(d) The lubrication of the mill should be safeguarded under the higher-temperature conditions.

(e) The tar addition should be made at the predetermined optimum time. It is usually in the range 5 to 8 per cent.

(f) Periodic checks of the packing density should be made in the A.F.A. rammer.

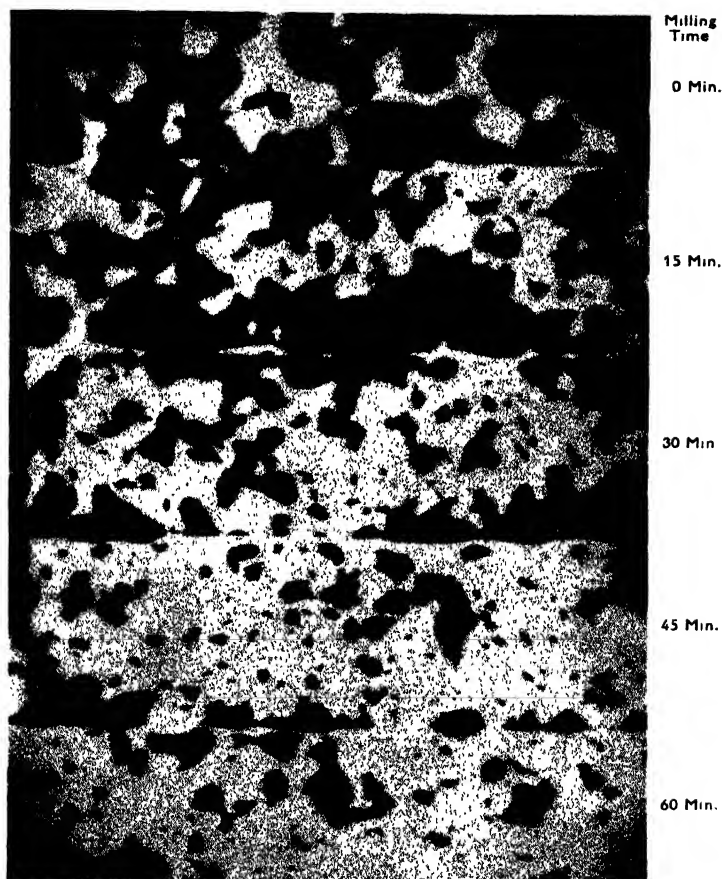
- (5) After tarring, the mixture should not be stored in a cold place for any extended period of time if subsequent difficulties in handling are to be avoided, and should be preheated to approximately 60°C. before ramming.

**(b) DRY-RAMMED DOLOMA HEARTHS**

In 1939 a British patent was filed by G. B. Crespi, an Italian steel-plant operator, for a method of installing dry furnace hearths using a mixture of particles described as being the size of rice and flour. Although the use of such dry gradings has for many years been standard practice in induction furnace linings, Crespi appears to have been the first to suggest the use of such finely ground dry material in the open-hearth furnace. He claimed that it could be readily installed merely by ramming with the relatively sharp tools (a discovery made many years previously in connection with induction furnaces), and that the presence of the fine fraction led to extremely low permeability and consequently to minimum penetration of the hearth by metal. Sufficient experience has been gained, particularly in Europe, with such hearths, to show that they are highly satisfactory and that the monolith does sinter to a singularly solid mass. Periodic claims that such hearths result in reduced fettling are not so well confirmed, the quality of the working surface of the hearth soon becoming more dependent on the fettling material used than on the nature of the original hearth. On the other hand "bad bottom time", which is normally associated with localised metal penetration may well be reduced.

Shortly after the Second World War a modified type of dry rammed hearth similar to the Crespi type but with a graphite addition was patented by Swift. Numerous hearths of this type have now been installed in Great Britain and appear to give similar service to the standard Crespi material.

One rather unexpected benefit from low permeability hearths, which was brought out rather forcibly after the Second World War, when many Continental steelworks lay idle for as much as 6 months, was the resistance of such hearths to deterioration by atmospheric hydration. Hearths that had been cold for as long as 6 months were still found capable of re-use.



*Fig. 170. Effect of milling time on intergranular porosity. Dark areas in contact print indicate pores in dolomite originally of  $\frac{1}{4}$ --7 mesh grading (after Cross, Marshall and Sarjant).*





*Fig. 171. Mobile felling machine throwing doloma through door on to back bank (courtesy Blaw Knox Ltd.).*

*Graded magnesite hearths with air setting bond*

For this purpose prepared grain size ramming materials are employed having dead-burnt magnesite as a base, or in some cases dolomite-magnesite mixtures. According to Sosman *et al.*, they usually contain not less than 60 per cent. nor more than 85 per cent.  $MgO$ , though mixtures as high as 96 per cent.  $MgO$  have been used. The ramming is done on top of basic brick between formers designed to permit ramming to the full depth in transverse sections while shaping the contour of the bottom. Correct consistency is obtained by adding water in a suitable mixer and then shovelling the mixture in layers of 3 in. to 5 in. thick—ramming being done by means of pneumatic hammers. The bottom must then be dried by means of multi-hole gas burners. When the main fuel supply is turned on the furnace is raised to about  $1600^{\circ}C.$  and held for, say, 24 hours before washing with slag and chilling. Incidentally this latter process, sometimes described as "setting back", is common in many parts of the world, and is supposed to confer a virtue on the hearth that is difficult to conceive in mineralogical terms. After reheating following chilling any cracks are filled in by fettling and the first run of material charged. In a modified procedure the lower part of the sub-hearth is made of plastic chrome ore, rammed over the basic brick to a depth of about 6 in. This is then burned for about 24 hours before being built up by fettling with magnesite. It is claimed that the use of this chrome layer protects the basic brick during the burning-in of the bottom and also provides a relatively impervious layer that prevents the penetration not only of steel but also of lead and other impurities introduced with the scrap.

Periodically a hearth is completely ruined during the installation process. Such tragedies can be due to a number of causes, *e.g.*, the use of old doloma that has already hydrated, water in the tar, or occasionally to the use of long stewing periods in the temperature range below  $200^{\circ}C.$  when the steamy atmosphere may well lead to hydration both of lime and magnesia, with consequent expansion and crumbling of the monolith. If for some reason a hearth cannot be put into service immediately after it is rammed it is better to allow it to stand for a day or so prior to heating up rather than maintain it for long periods at temperatures around  $100^{\circ}$  to  $200^{\circ}C.$

*Fettling*

The success obtained with a B.F.L.C. type hearth led to an almost equally intensive search for the ideal fettling material. Trials were first made using extended gradings similar to those employed for the hearth

but without tar additions. These were found to suffer from one very serious limitation, *viz.*, a strong tendency to segregate during throwing, the coarse material arriving first and a considerable proportion of the fines being blown by the flame into the downtakes. Numerous attempts were made to overcome this difficulty, for example, by the addition of small amounts of tar, oil and even water, but the final conclusion reached was that the most economic method at present available was the use of a material of a relatively close grain size range and almost completely free from fines. The recommendation that as far as possible the doloma used should be hard-burned material (bulk density greater than 2.5 g.p.ml.) and of a grading essentially 100 per cent.  $\frac{1}{2}$  in.-72 mesh, was supported by a substantial amount of work on the effect of grading on angle of rest—a particularly important property where steep slopes must be maintained. It was shown that some fines could be carried with advantage if 2 per cent. of tar were added but this practice does not appear to have found any very wide application.

Another recommendation of this Committee, *viz.*, that fettling should be done with gas on, has been by no means generally adopted. During the Second World War operating conditions were so strenuous, due to the necessity for blackout of melting shops, that the practice of fettling with the fuel off became fairly general. Many firms still continue this practice, and there is little doubt that both the quantity of doloma used and the fettling times are substantially increased. In the United States where the long haul between dolomite and steel plants is liable to result in the hydration of dead-burned doloma, as used in Great Britain, there is a tendency to resort to the use of proprietary brands of dolomite clinker to which substantial iron oxide additions have been made—normally in a rotary kiln. For bank fettling, raw dolomite, which is cheap and stable, is frequently employed, although there is little doubt that it is a very inferior material to well-shrunk doloma. Magnesite is rarely used for general fettling, though it is frequently employed for repairs to holes in the hearth. The relative economics of dolomite and magnesite are very much dependent on both steelmaking practice and local prices. In Austria, where magnesite is abundant, such hearths have long been employed, whereas in Great Britain even with domestic sea-water magnesia available, the use of well-fired doloma is generally more economic both for bottom making and for fettling. A possible exception occurs in plants making particularly low carbon steels, where the best possible hearth is needed in order to cut down both fettling consumption and fettling time.

Mention should be made of the use of mechanical devices for fettling, the most common being the Blaw-Knox type machine illustrated in

fig. 171, p. 394. This enables large quantities of dolomite to be shot on to the banks in only a fraction of the time required by hand shovelling and with much less strain on the operators. Certain fettling, *e.g.*, of holes and of front wall banks, must, however, still be done by hand. Holes in hearths present a particularly difficult problem, frequently requiring periods of many hours to repair. The normal practice was, and in many plants still is, to splash out any metal or slag from the hole by means of wooden rabbles. This very arduous and by no means always effective method has given place in many plants to blowing out of the metal with a jet of compressed air.

The actual consumption of dolomite for maintenance of furnace hearths varies greatly according to the metallurgical load and furnace type, figures ranging from 40 lb. to 120 lb. per ton of steel being quite usual. The lowest values are obtained by large tilting furnaces, where the continuous process rarely results in the full hearth being exposed, whilst the highest consumptions arise in plants having a cold charge and engaged in the production of particularly low carbon steels. Where magnesite is employed the poundage used may be only one-third or less of the above figures, but even so the total cost may well be greater.

### (c) LIFE AND CAUSES OF FAILURE

It was at one time quite customary to hear open-hearth men boast of the long lives of their basic open-hearth bottoms, periods of 15 to 20 years often being claimed. Anyone making such a claim today is liable to be accused of poor practice, since it is now realised that to keep a bottom in for such a long time is likely to be uneconomic. The reasons for this change of view and the case for renewing a dolomite hearth after, say, 2 years, can be summarised as follows:

- (1) So-called bad-bottom time, *i.e.*, the time taken by excessive repairs of say 2 hours or more, tends to increase markedly with bottom age.
- (2) Fettling time goes up quite substantially even within the first year of life, as is for example illustrated in fig. 172. As might be expected fettling consumption goes up in a somewhat similar manner. The illustration, which refers to average data plotted week by week for a cold-charged 80-ton furnace making low carbon steels, shows a characteristic fluctuation which sometimes becomes violent, but a definite increase in fettling time from about 50 minutes for a new hearth to about 80 minutes for one that is only a year old. Although the increase in the year following is likely to be less, the incidence of bad-bottom time is greater.

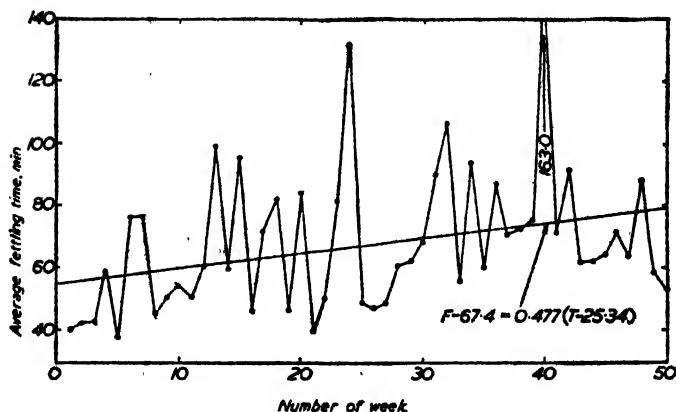


Fig. 172. Change in fettling time with age of hearth.  $F$  = fettling time in minutes,  $T$  = age of hearth in weeks (after Brown and Ward).

- (3) The risk of breakouts increases due to metal penetrating to unknown depths in the hearth and the imperfect cleaning out of holes in the bottom.

The change to the more rapid replacement was a natural outcome of the development of techniques for rapid bottom replacement, it being readily shown that the amount of dolomite (or magnesite) used in a new hearth when spread over, say, two years, is negligible in comparison with the increase in fettling consumption associated with an unsatisfactory hearth.

Much has been written regarding the changes which occur in both dolomite and magnesite hearths in use and the part which the hearth plays in the chemical actions occurring in the bath. It is known, for example, to contain a substantial amount of iron and iron-oxide, which absorbs oxygen during the fettling period and subsequently gives it up by reaction with metalloids in the bath. The main causes of deterioration are:

- (1) The absorption of fluxes, principally iron oxide but also lime and sometimes silica, from the charge
- (2) Metal penetration through cracks or weak spots in the hearth
- (3) Loss of the original shape

Numerous authors have described the changes which occur, in bottoms, e.g., those in dolomite hearths have been discussed in considerable detail in the B.F.L.C. Report (I.S.I. Special Report No. 33), both for cold and hot metal practice. Samples were, for example, taken from an 80-ton furnace at Messrs. Steel, Peech and Tozer, after 3 years operation Fig. 173, p. 403, shows a section through such a hearth, including the

TABLE XXXII  
ANALYSES OF SAMPLES REMOVED FROM VARIOUS DEPTHS IN "F" FURNACE BOTTOM,  
TEMPLEBOROUGH

Sample No. 1 represents the working face, and No. 5 the unsintered portion

Sample No.	SiO <sub>2</sub> %	CaO %	MgO %	MnO %	S %	P <sub>2</sub> O <sub>5</sub> %	FeO %	Fe <sub>2</sub> O <sub>3</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Loss %
1	6.56	51.6	19.5	1.99	0.27	2.47	10.75	4.42	1.13*	—
2	3.80	50.8	18.2	0.39	0.60	3.42	13.09	2.12	1.46*	—
3	3.18	51.7	14.0	0.17	0.81	1.42	11.45	2.29	10.08*	—
4	3.20	51.2	16.8	0.21	1.06	1.21	11.40	1.79	8.15*	—
5	3.32	55.2	30.1	0.19	0.76	0.98	1.14	0.04	1.25*	3.04

\* Analysis made on similar but not identical samples.

relatively thin sub-hearth of basic bricks. Tables XXXII and XXXIII, shows a series of chemical analyses, bulk density, specific gravity and porosity measurements, made on zones removed from this hearth, sample No. 1 being a top 3 in. of hard and glassy looking material, in which the original grading is no longer visible, and sample No. 5 containing practically unaltered doloma. This latter was weakly sintered and perished readily on hydration. A study of these tables shows that the principal contamination is due to iron-oxide, the final

TABLE XXXIII

PHYSICAL PROPERTIES OF SAMPLES REMOVED FROM  
"F" FURNACE HEARTH, TEMPLEBOROUGH

<i>Sample No.</i>	<i>Bulk density (Mercury displacement) G. per ml.</i>	<i>Specific gravity (Rees-Hugill flask)</i>	<i>Porosity (calculated) %</i>
1	3.45	3.48	0.8
2	3.04	3.38	10.1
3	3.30	3.39	2.7
4	3.15	3.37	6.5
5	2.09	3.33	37.2

content of which is of the order of 15 per cent. in the top layers, compared with only about 1 per cent. in the raw material. The drop in lime is relatively small, whilst a surprisingly high decrease occurred in the magnesia content. There is also an appreciable absorption of phosphorus as might be expected in view of the fact that the hearth is periodically bathed with basic slag containing 5 per cent or more of  $P_2O_5$ . The porosity figures show that an abnormally high value in the bottom zone has changed to less than 1 per cent. for the top zone, even the figure of 10 per cent. for the second probably being due in the main to the presence of rather large cavities. X-ray examination of these same samples shows that magnesia and lime, which are virtually the sole constituents in the raw material, are also the principal ones present after use, but that their lattice spacing has changed due to the absorption of appreciable amounts of  $FeO$ .

The changes undergone by magnesite hearths were first discussed in some detail by Andersen, who showed that the periclase absorbs large amounts of iron oxide, the proportion present in the upper layers of an old bottom being as great as 50 per cent. Andersen also showed

substantial recrystallisation at the working face, the periclase grains gradually joining up to form an almost continuous network, the interstices of which were filled with fluid silicates and calcium ferrite. The problem has been studied more recently by Kraner and by Snow, the former having reported quite fully both on the phase reactions likely to occur in hearths and on examination of core samples drilled both from new and old magnesite hearths. Even the unused hearths showed a magnesia content of about 70 per cent. due to appreciable dilution with slags of high lime/silica ratio and high lime content used to facilitate burning-in. From the data given in Table XXXIV from Kraner's paper it will be seen that much of the magnesia had been removed and that which remains is contaminated by other oxides, particularly lime, silica and iron oxide. Incidentally it will be seen that this particular hearth was formed over a chrome plastic and chrome brick sub-hearth, which do not appear to have changed much in service. Samples taken from another position, *viz.*, in front of the tap-hole, showed that all the magnesite had been replaced by dolomite, the top 2 in. having a lime content of 38 per cent. and a magnesia content of only 24 per cent. Here again iron-oxide was found in very substantial quantities.

#### (d) LINES OF IMPROVEMENT

Given an adequate incentive markedly improved hearths could doubtless be produced, *e.g.*, by employing cast magnesia blocks, but within the ambit of present practice the following points would most repay attention:

- (1) The production of fully rammed hearths having even higher packing densities, by the choice of suitably graded material of high sintering tendency.
- (2) Improved drainage of the hearth, by maintenance of correct contours and a high temperature for tapping—particularly in the taphole region.
- (3) The use of specially graded and bonded fettling material showing a greater angle of rest, less segregation on throwing and consequently a higher effective packing density.

#### TAPHOLES

The taphole, which is normally located in a position vertically below the centre of the backwall, and at a point lower than any other on the hearth, can be made in a great variety of ways. The most common one is to leave a relatively large hole in the magnesite or stabilised dolomite brickwork and ram the gap between this frame and a wooden plug



TABLE XXXIV  
HOLE NO. 2—NO. 36 FURNACE—MAGNESITE HEARTH  
Base of End Slope, Centre of Furnace  
April 1938 (*After Kraner*)

Porosity and Sp. G.				Chemical analysis										
Material	Poros.	App. G.	Bulk Sp. G.	Depth of Sample	MgO	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	FeO	Fe <sub>2</sub> O <sub>3</sub>	Metallic Fe	Ignition Loss
Magnesite	6.84	3.46	3.23	Top-1 1/2"	56.9	15.2	9.2	3.3	0.04	1.2	9.0	2.3	0.7	0.5
"				1 1/2"-2 1/2"	62.3	13.6	10.4	2.5	0.03	0.9	5.9	0.6	0.4	1.6
"	7.75	3.42	3.16	3 1/4"-4 1/2"	65.8	11.8	8.8	2.0	0.04	1.2	5.9	0.7	0.7	1.4
"				4 1/2"-5 1/2"	65.5	11.8	9.7	1.9	0.24	1.1	5.9	1.1	0.4	1.3
"	8.93	3.60	3.27	6 1/2"-7 1/2"	24.8	5.2	12.8	10.7	29.3	—	13.5	—	0.3	0.8
Chrome plastic				7 1/2"-8 1/2"	16.8	5.1	14.0	10.9	35.7	—	12.5	—	0.3	2.2
"				8 1/2"-9 1/2"	16.6	1.1	16.8	12.6	36.2	—	12.4	—	0.4	0.4
"	7.81	3.57	3.29	9 1/2"-11 1/2"	13.7	Trace	17.4	14.6	37.3	—	13.1	—	0.2	0.2
"				12 1/2"-13 1/2"	13.1	"	12.2	13.1	39.3	—	14.3	—	0.3	4.0
"	15.50	3.66	3.10	13 1/2"-14 1/2"	12.9	"	12.2	12.9	39.0	—	12.6	—	0.2	6.2
Chrome brick				15 1/2"-16 1/2"	17.0	"	8.9	20.4	36.5	—	13.3	—	0.1	2.1
"	12.25	3.45	3.03	16 1/2"-19 1/2"	17.1	"	9.4	20.4	36.3	—	13.8	—	0.1	1.1
"				19 1/2"-19 3/4"	17.3	"	8.5	22.0	36.5	—	13.6	—	0.1	None



*Fig. 173. Vertical hearth section from cold-charged open-hearth furnace, showing monolithic doloma lining and 9 in. stabilised dolomite sub-hearth (after Bradley, Chesters, Ferguson and Sarjant -- I.S.I. Special Report No. 33).*



*Fig. 174. Tapping of large tilting type open-hearth furnace.*

with a suitable basic cement, *e.g.*, magnesite, stabilised dolomite, or one of the various proprietary brands containing chrome ore. When the furnace goes into operation, the wooden core burns out leaving a hole, which is then filled with a mixture of burnt dolomite and anthracite. When the furnace is ready to tap the loose dolomite on the outside is dug away and an oxygen lance used to burn through the last section until the metal comes. More recently use has been made by certain firms of an explosive charge set in the taphole and detonated from a distance. This is stated to provide a clean hole of a rather larger size than is normally first formed by oxygen lancing and thus permit of a quicker and more consistent tapping of the heat.

### THE LANDER

The steel normally runs out of the taphole, *see* fig. 174, opposite, on to a chute (known as a lander or launder), which is usually lined with fireclay brick coated with "ganister". Since the ganister has to be renewed with every heat, and quite a serious cut occurs, it might well appear that such a practice would lead to dirty steel. In all probability, however, most of the ganister introduced into the ladle rises to the top and remains in the slag rather than being trapped in the steel. Attempts have been made to improve steel cleanliness by the use of basic bricks in the lander, but the general practice still appears to be the use of a siliceous-lined chute. The latter may be of the simple type, or where it is desired to tap the contents of a large furnace into two ladles, may be bifurcated, the final level in the two ladles being kept the same by deviating the stream to one side or the other by means of a wooden paddle.

Where "ganister" (clay-bonded quartzite) is employed it is essential that it be prepared with adequate control, since any wide variation in the grading, clay content, or moisture content, may lead to trouble. If the clay or water content is too high, abnormal shrinkage may occur and the ganister crack and come away from the firebrick backing, while if the grading is abnormally coarse it will be too gritty to trowel well on to the firebrick chute. A typical lander batch is given in Table XXXV. Almost any good ganister or quartzite is suitable, provided it is properly ground and indeed it is doubtful whether the use of really high-grade material is justified for this purpose. Sandstone, however, is not satisfactory, as it yields a very high proportion of 72–150 mesh material when ground. Even with the normal clay addition such gradings have little green strength. In works where the same ganister is employed in more severe positions, *e.g.*, in the tapholes of acid furnaces, the best material is, of course, essential. In one works the

grading of the batch is controlled in a very simple way, the grinders being provided with a spring balance, a half-pint cup, and a 7 (B.S.I.) mesh sieve. The material is fed into a solid bottom pan mill, together with a suitable amount of water, and ground until a certain weight of material remains on a 7 mesh sieve from a cupful of sample. The gradings given in Table XXXV, which were taken at weekly intervals over a period of a month, show that remarkably consistent results can be obtained in this way.

The lander is dried out prior to use either with a coal fire or with coke oven gas flares and is usually purged after each cast.

## THE GAS AND AIR UPTAKES

*(Sections 2 and 4)*

### (a) CONSTRUCTION

In open-hearth furnaces using producer or mixed gas, the fuel is normally brought from the slag pocket to the furnace chamber by means of a simple rectangular duct, which on an 80-ton furnace is about 2 ft. 6 in. or 3 ft. square. If the duct is too small a serious pressure drop occurs, whilst if it is too large there is unnecessary surface exposed for heat losses.

Far more important are the shape and location of the air uptakes, which on both gas and liquid fuel fired furnaces exert a major influence on the air distribution within the furnace, and therefore on the mixing and combustion patterns. By far the largest number of gas and driven fuel furnaces have twin uptakes at each end, generally in the form of rectangular openings in the corners of the furnace ends on either side of the gas port or doghouse. With this design the air stream rises vertically until it hits the port roof and enters the flame from the top and sides, being thrown down on to it in some furnaces by means of a knuckle between the port and main roof and in others merely entrained by the momentum of the fuel stream. Gas-fired single-uptake furnaces are comparatively rare, probably because their use leads to certain complications, notably the tendency of the uptake itself to get overheated due to the absence of a substantial temperature drop at the incoming end. This latter in turn appears to be due to the fact that combustion starts earlier and radiation from the flame itself passes vertically downwards on to the uptake walls. With driven fuels it is usual to push the burner a substantial distance into the furnace, so that relatively little if any of the flame is vertically above the uptake, although the air entrained at the root of the jet is still supplied directly from it instead of being entrained after passage to the port roof and back.

TABLE XXXV  
BATCH COMPOSITION AND GRADING OF LANDER GANISTER

		Tons				Cwts.	
Lander ganister batch:							
Rock ganister (97 per cent. $\text{SiO}_2$ )		..	..	..	..	6	0
Used roof bricks		..	..	..	..	—	15
Siliceous clay		..	..	..	..	—	10
<i>B.S.I. sieves</i>		<i>Nearest equivalent Tyler sieves</i>		<i>Standard grading</i>		<i>Typical gradings for 1 month</i>	
				%		%	
On 7 mesh		On 8 mesh		15		18.6	
7-25 mesh		8-28 mesh		30		28.4	
25-72 mesh		28-65 mesh		10		7.8	
Through 72 mesh		Through 65 mesh		45		45.2	
				%		%	
				21.2		18.4	
				25.8		30.2	
				8.4		7.4	
				44.6		44.0	
						16.4	
						29.6	
						5.4	
						48.6	

In twin-uptake furnaces two rectangular flues, say, each 6 ft.  $\times$  3 ft. on an 80-ton furnace, are employed, but with single-uptake design there is a tendency to use rectangular or oval-shaped uptakes with their long axis at right-angles to the flame direction. This arrangement enables the burner tip to be brought relatively close to the bath without an undue length being exposed to the furnace gases. The design of the uptakes is, however, equally important in relation to exit conditions and one advantage claimed for the single uptake design is that it tends to draw the waste gases off the side walls and roof, whereas the twin-uptake tends to pull them into the corners with consequent impact on the splay walls. The relative areas and lengths of the ducts are also important, particularly with gas-fired furnaces, where too small an area on the gas side may lead to relatively little of the waste gases leaving the furnace through this duct and consequently a relatively low preheat on the reverse cycle.

Attempts to minimise wear on the exit end, by sloping it outwards towards the bottom, have given mixed results, some operators claiming a big improvement whilst others have found little change. The lack of any marked improvement in some designs is due to the fact that at least in the top section of the uptake there is a tendency for the waste gases as they turn through a right-angle bend to eject the coarser flux drop-lets regardless of whether the uptake is vertical or sloping. On the other hand the use of such a sloping end does appear to be an advantage as regards roof wear, since it leads to a cleaner sweeping of the incoming end with air.

Several plants have experimented with suspended construction in uptakes during recent years, for example several furnaces have been built with complete basic ends of the Detrick suspension type. That such techniques have their value in maintaining critical arches, such as those at the entrance of the slag pockets, cannot be doubted, but for normal vertical wall construction their use is doubtful, since it generally results both in a relatively open structure due to the lack of broken joints, and increased difficulty of repairs.

#### (b) MATERIALS

In the past uptakes were normally built of good quality silica brick and only required frequent repair in the top or target section. With faster driving, however, the duty imposed on the uptakes is far greater both because the velocities and consequently probably the amounts of flux carried are higher, but also because exit temperatures tend to rise with higher fuel rates. For this reason there has been a tendency to go

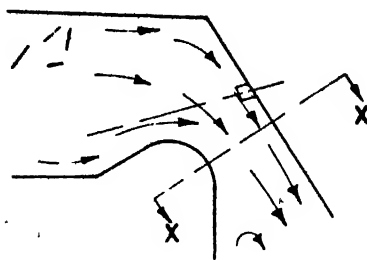
to basic construction either for the whole of the uptake or at least for the pit-side section, which due to asymmetry in the exit flow normally works under the most severe conditions. Good results have been obtained with both fired and unfired chrome-magnesite and magnesite-chrome bricks in this position, whilst remarkable lives have also been achieved with downtakes built from metal-cased magnesite bricks.

### (c) LIFE AND CAUSES OF FAILURE

At one time it was usual for both the air and gas uptakes on an 80-ton producer gas furnace to have a life of the order of a year, with a corresponding brick consumption of only 1 to 2 lb. per ton of steel produced. With faster driving, however, repairs tend to be far heavier, and have even proved necessary with basic bricks (at stage level) after, only, 8 weeks. The gases leaving the furnace are probably higher in flux content than anywhere else in the furnace and the coarser droplets are probably ejected for the most part during the first bend. Furthermore the wall being vertical any reaction products tend to flow down the surface on to the bricks below, attacking them before dripping into the slag pocket. The flow in downtakes is rarely if ever simple, water models indicating that with square section downtakes the flow is generally in spiral form (as with water leaving a bath tub) whilst with elongated uptakes at right-angles to the exit direction twin eddies may be set up as a result of secondary flow (fig. 175), leading to corresponding areas of wear on either side of the centre line (fig. 176, page 413). Analyses of dust caught on water-cooled probes pushed horizontally into the uptakes show it to consist in the main of iron-oxide with lesser amounts of lime. The actual dust content varies greatly during the heat, reaching a maximum shortly after scrap charging and before the roof temperature reaches its maximum level. Furthermore, the amount of fume varies greatly with the process, *e.g.*, it would appear to be much higher with the use of pitch-cresote as the fuel than with oil and higher for low steam/oil ratios than for high steam/oil ratios.

With silica bricks attack would appear to be a straight forward lowering of the melting point by absorption of fluxes at a temperature reaching  $1700^{\circ}\text{C}$ . and an atmosphere that can also periodically be reducing even though combustion control is set for 2 per cent. oxygen in the waste gases. With basic bricks, wear is more usually due to spalling, though how much of this is due to iron-oxide bursting and how much to the structural spalling associated with differences in properties between the slagged surface and the unslagged back is by no means clear.





VERTICAL SECTION THROUGH C



• INDICATES DOWNWARD FLOW

*Fig. 175. Flow pattern in exit end of single-uptake furnace showing secondary flow.*

#### (d) LINES OF IMPROVEMENT

- (1) Some improvement might be obtained merely by changing the shape of uptakes, *e.g.*, from square to a round section, or from a rectangle to an oval, though with obvious complications in construction. It is more likely, however, that progress from the design standpoint will come by consideration of flow in the system as a whole, leading for example to reduced vorticity in this part of the system.
- (2) Although the longest lives are at present obtained with basic bricks of a type intended for use in basic roofs, it may be that the somewhat different conditions and in particular the relative lack of arch stresses may favour the use of extremely dense bricks even though density is obtained by sacrifice of refractoriness.

## GAS AND AIR SLAG POCKETS

(Sections 17 to 25)

## (a) CONSTRUCTION

In the early Siemens' furnaces the gases leaving the melting chamber passed directly into the checkers. There are still a few furnaces of this type in existence but they have long since been considered obsolete due to the slagging and choking of the checker filling by the large amount of carry-over. Ideally the slag pockets should be designed as cyclone separators, but in most furnaces they are just a rectangular box having a loose brick lining separated from the main chamber walls by a filling of refractory sand (*see* fig. 177, p. 413). As the campaign continues the pockets fill up, generally with material that is molten at the top surface but relatively solid lower down. More recently attempts have been made to tap the molten slag periodically from the pockets and thus extend their life beyond the usual 13 or so weeks after which a shut-down to remove slag usually becomes necessary. Until comparatively recently the large block of slag so formed had to be removed by hammer and bar techniques with a consequent long delay. More effective methods have, however, been developed—principally in the United States—and are now finding wide acceptance. Sosman *et al.*, summarise these as follows:

- (1) Mechanical breakers, such as a beam suspended by the pit crane and actuated by the weight of a heavy object such as a ladle. After being broken, the slag is removed by wire ropes and pulleys or by trucks equipped with scoops.
- (2) Explosives, which may be placed in holes drilled in the slag at the completion of the campaign or in tunnels formed by firebrick sleeves or bottom-pour brick placed in the pocket during the previous reline.
- (3) Slag cars—flat-topped cars or sleds that are designed in conjunction with the false walls of the pocket so that the slag can be pulled out without preliminary breaking.

In many modern furnaces and in particular in those which are built all-basic, or with completely basic ends, the arches in the slag pockets are built with suspended construction, *e.g.*, of the Detrick or Laclede-Christy type.

## (b) MATERIALS

With slow rates of drive second quality silica bricks were considered quite adequate for slag pocket chamber walls and arches. With increasing fuel rates, however, disastrous failures have occurred with inferior

bricks and the use of first quality silica brick of the type used in open-hearth furnace roofs is, therefore, recommended, at least for the arches. The second quality brick should however be satisfactory for the false walling and moderate quality sand with not too high a sintering tendency for the separating layer. Used bricks are frequently employed for the construction of the bottom paving, whilst the slag wickets can also be built of relatively low grade brick. On gas-fired furnaces the risk of failure in the wall separating the gas and air sections can be greatly reduced by using air cooling boxes between the two pockets.

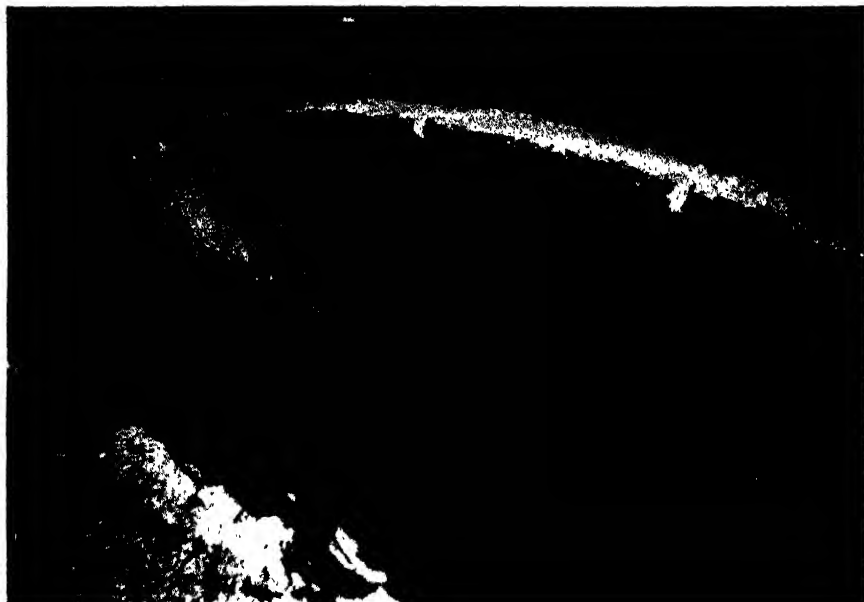
### (c) LIFE AND CAUSES OF FAILURE

The conditions responsible for damage to slag pocket refractories are essentially similar to those in the uptakes, though the temperatures are normally about 200°C. lower. Figures of nearly 1550°C. have, however, been registered and might well be exceeded where trouble was experienced, say, with a furnace valve and reversal therefore delayed. The type of material formed in the slag pocket on a silica end furnace is shown by the analyses given below:

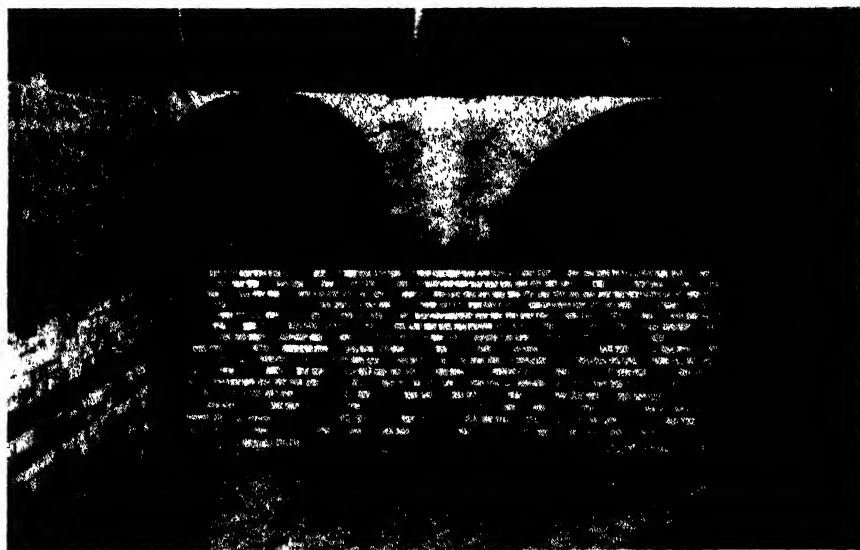
			<i>Analysis of slag from:</i>	
			<i>Air slag pocket</i>	<i>Gas slag pocket</i>
SiO <sub>2</sub>	..	..	46.9 per cent.	47.2 per cent.
Total Iron as Fe	..	..	30.6	25.1
Al <sub>2</sub> O <sub>3</sub>	..	..	1.3	1.6
MnO	..	..	1.0	1.1
CaO	..	..	9.2	13.9
MgO	..	..	2.1	3.4
SnO <sub>2</sub>	..	..	0.1	0.1
PbO	..	..	0.8	0.7
CuO	..	..	0.1	0.1
ZnO	..	..	2.2	1.6

From which it will be seen that the slag consists essentially of silica and iron oxide, together with roughly one-third as much lime as iron. Such a mixture has a relatively low melting point and can normally be tapped at the maximum temperatures achieved during the refining period.

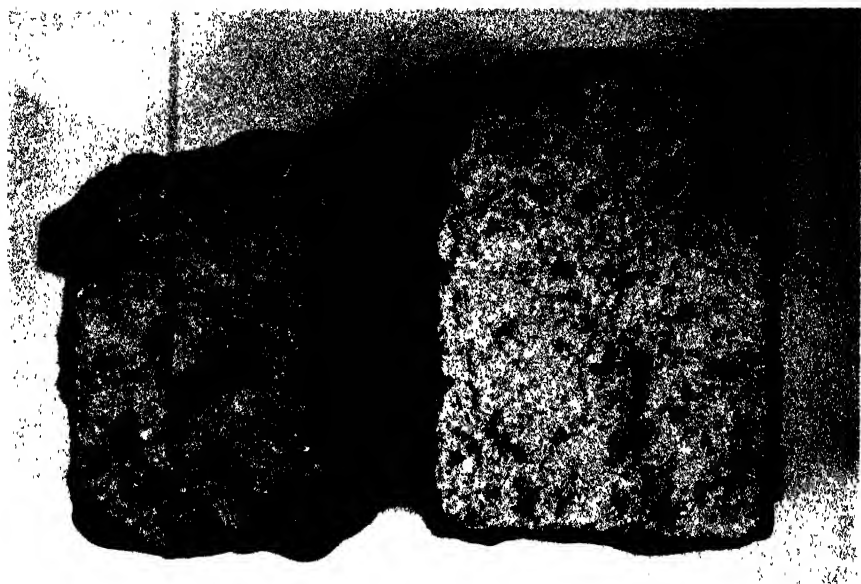
Since it is mainly the result of reaction between iron and lime brought over from the furnace and the silica end walls, it is not surprising that a very different material is found when basic end construction is employed. With such furnaces a far more refractory deposit occurs, consisting mainly of spalled basic brickwork mixed with iron oxide and again a proportion of lime. In some furnaces this deposit is so crumbly



*Fig. 176. View of single-uptake below stage level showing wear believed due to secondary flow.*



*Fig. 177. Large slag pocket on single-uptake furnace—twin checker chambers retained.*



*Fig. 178. 42% alumina firebrick (left) and 55% alumina brick (right) after 18 weeks' service in an open-hearth furnace checker.*

that it can actually be raked out, but in others where it has sintered together it is so tough as to be at least as difficult to remove as the fully fused product found with silica ends.

(d) LINES OF IMPROVEMENT

- (1) The principal aim should be to increase the efficiency of dust removal by getting a better centrifuging action. Model work already suggests possibilities, since the flow pattern given by a single uptake entering the slag pocket centrally is a twin vortex, whilst that with one entering tangentially is a much larger single vortex. Given methods of periodic tapping of slag the original shape could be retained and, therefore, the initial efficiency as a dust collector.
- (2) The techniques for slag removal widely employed in the United States might well be extended with advantage to plants elsewhere.
- (3) The rate at which the slag pocket fills up could be substantially reduced, both by the use of basic ends and by avoiding the pushing of bricks from the target area into the slag pocket during running repairs.

## CHECKER BRICKS

(Section 33)

Anyone who has tried to melt steel scrap without the use of preheat (or oxygen enrichment) will appreciate the big advance made by Siemens when he designed a reversing type furnace fitted with a system of heat regeneration. The use of checkers yields a double advantage about half the heat is extracted from the waste gases leaving the furnace and when returned to the system in the form of preheated air (and with gas furnaces preheated gas), it results in a substantially higher flame temperature and, therefore, a faster melting rate. The current practice both as regards the materials used for checkers and the particular settings employed varies widely from one works to another, and little reliable information is available regarding the relative efficiencies of the different arrangements. What is known is that too open a setting gives a low thermal efficiency and inadequate preheat, whilst too tight a setting rapidly leads to choking of the flues by flux deposition. The lack of reliable data is not surprising when the difficulties of carrying out a full heat balance are considered, whilst the alternative approach of calculating efficiencies on a fundamental basis is even more difficult and certainly less reliable, since the conditions assumed only refer to those existing in the early stages of a campaign. The most vital need at

the moment is for reliable data on temperature distribution within different types of checker settings and the effect of these on preheat temperatures at various stages in the campaign. Incidentally the air preheat as measured in the uptakes at stage level is generally about 150°C. higher than that at the checker bridgewall due to the additional heat transfer which occurs between the turbulent air stream and the extremely hot surfaces in the slag pockets and uptakes.

#### (a) CONSTRUCTION

A bewilderingly large number of checker settings have been designed, many of which have been patented. Numerous advantages are claimed, in particular extra heating surface, maintenance of high gas velocity in the lower part of the chamber, freedom from choking and high stability. The fact that steelplants frequently change their type of settings, for example from the normal basket weave to special shapes, and later to different types of basket weave or solid chimney settings, frequently finishing with the original arrangement, shows that in practice the differences between these types is not as great as claimed, partly because they are all modified in practice by the corrosive and choking action of the waste gases.

Calculation shows that substantial differences can exist for example in the vertical heating surface but that these are frequently offset by lower weight per unit volume, or in actual life by reduced stability leading to collapse, at least of the top courses.

Another unknown which must have a marked effect on operating efficiency is the distribution throughout the setting, both of the waste gases on the exit end and the air on the incoming end. Even water model studies may fail to give a correct appreciation of such distribution, since superimposed on the flow to be expected from a checker at uniform temperature there are differences in buoyancy lift from one end to another according to the temperature of the particular "chimneys" concerned. The suggestion that comparisons should be made by filling one checker chamber with a given type of setting and that at the opposite end with another type, although academically having much to commend it, suffers from the disadvantage that any small difference would be completely swamped by other out-of-balance factors, whilst any major difference would prove embarrassing to the operator.

#### (b) MATERIALS

##### (i) *Fireclay and semi-silica brick*

Most open-hearth furnaces in Great Britain still employ either medium alumina (say 35 per cent.) or semi-silica (sand-clay bricks) for

the bulk of the checker work, but use silica or high alumina (40 to 42 per cent.) fireclay brick for the top 5 to 10 courses. Test data for such bricks are given in Table XXXVI.

*Porosity and bulk density:* In selecting checker bricks more attention should be paid than hitherto to these factors. Present-day checker bricks frequently have porosities in the range 20 to 25 per cent. or over, even though porosities as low as 13 per cent. and bulk densities as high as 2.25 g.p.ml. are obtainable.

*Specific heat:* Refractory materials vary less in this property than almost any other, the value both for aluminosilicate and silica bricks being generally close to 0.25.

*Thermal conductivity:* As Austin has shown the thermal conductivity of a test brick is very much a matter of porosity and if the latter be kept low high conductivities are likely to be obtained, particularly with well-fired bricks. Values obtained for the fireclay and semi-silica bricks as shown in Table XXXVI must all be considered as of the same order.

*Heat capacity and diffusivity:* The heat capacity of a checker brick—assuming it to be of uniform temperature throughout—is a direct function of the bulk density and specific heat. The product of these terms, calculated for the temperature range 750° to 20°C., is close to 0.50 both for fireclay and semi-silica brick. The rate at which heat diffuses into the brick depends both on the thermal conductivity and on the heat capacity, the diffusivity factor [= thermal conductivity ÷ (bulk density × specific heat)] being much the same for the various bricks considered. Although the above observations are approximate and only refer to selected samples, they do suggest that the difference in thermal behaviour between silica, semi-silica and fireclay bricks are more a function of the particular brands and, therefore, for example, of differences in porosity rather than of chemical composition.

*Other properties:* The importance of other properties, e.g., melting point, depend greatly on the temperatures likely to be found in the particular settings. These are normally substantially higher in British than in American furnaces, the latter frequently operating with a maximum temperature at the top of the setting of the order of 1100°C. (2012°F.) compared with nearer 1400°C. (2552°F.) in many British furnaces. That this latter temperature is not unusual was brought out by some recent surveys, in which checker top temperatures in excess of 1550°C. (2822°F.) were recorded. Under such conditions some of the bricks would approach their melting point even if they were not being slagged by substantial amounts of iron oxide, lime, alkalis, zinc, lead and other fluxes.



TABLE XXXVI  
THE PROPERTIES OF CHECKER BRICKS

	Semi-silica			Fireclay	Silica
	(1)	(2)	(3)		
Chemical analysis:	%	%	%	%	%
SiO <sub>2</sub> .. .. .	88.7	92.3	88.0	*53.8	95.8
Al <sub>2</sub> O <sub>3</sub> .. .. .	9.1	5.8	7.9	39.2	0.8
Fe <sub>2</sub> O <sub>3</sub> .. .. .	1.0	0.5	1.2	2.7	0.9
TiO <sub>2</sub> .. .. .	0.4	0.7	0.7	1.6	0.0
CaO .. .. .	0.2	trace	0.4	0.3	1.6
MgO .. .. .	0.3	0.2	trace	0.7	0.3
Alkalis .. .. .	0.4	0.5	0.8	1.4	0.5
Loss on ignition, per cent.	—	—	0.3	0.2	0.2
Refractoriness, °C.	over 1600°	over 1600°	over 1600°	1560°	1600°
Cold crushing strength, lb. per sq. in. ..	1480	over 8030	1250	2350	4680
Porosity, per cent.	23.6	23.3	27.7	26.3	26.8
Bulk density, g.p.ml. .. .. .	1.93	1.90	1.85	1.96	1.70
Thermal conductivity (hot face, 700°C.):					
B.Th.Units .. .. .	5.6	7.1	5.6	6.2	7.5
C.G.S.Units .. .. .	0.0019	0.0024	0.0019	0.0021	0.0026
Specific heat, c.g.s. units (from 750°C.)	0.26	0.245	0.27	0.25	0.255
Heat capacity = specific heat × bulk density	0.50	0.47	0.50	0.49	0.43
Average .. .. .	0.49			0.49	0.43
Diffusivity factor = $\frac{\text{Thermal conductivity}}{\text{Bulk density} \times \text{specific heat}}$	3.8	5.1	3.8	4.3	6.0
Average .. .. .	4.2			4.3	6.0

\* Supplier's analysis.

*(ii) Silica bricks*

Silica bricks have been found more resistant to slag and fluxes at the top of checker settings than ordinary 35 per cent. alumina bricks and in some plants are actually used throughout the checker setting.

*(iii) High alumina bricks*

Bricks in the 42 per cent. alumina class and of high bulk density have given excellent service in the top courses of checker settings and have been standardised at certain plants partly because of their somewhat higher resistance to fluxes rich in zinc oxide. More recently the excessive temperatures experienced in fast driven furnaces of relatively small checker volume has led to trials of 55 per cent. alumina bricks made with an andalusite base. In the initial tests such bricks remained relatively unattacked after the semi-silica bricks in the lower courses had completely collapsed, whilst in subsequent trials where higher grade bricks were used in the lower courses the 55 per cent. alumina part was good enough to go back into operation after a campaign of over 40 weeks. Marked differences in rate of attack on 55 and 42 per cent. alumina brick at the same level in a checker is illustrated by fig. 178, p. 414, which shows their condition after 18 weeks operation.

*(iv) Basic bricks*

For several years now extensive use has been made of basic bricks in glass tank checkers—the alumino-silicate bricks used previously having suffered from certain limitations, notably shelling due to the formation of a complex alkali alumino-silicate. More recently trials have been made both in the United States and in Europe of such bricks in steel-plant furnaces, the results of which have been sufficiently encouraging to warrant extended use. This trend is one which is likely to continue, since faster driving of existing furnaces inevitably leads to higher checker top temperatures, whilst the realisation that preheat is a vital factor in driving rate encourages the operator to use checker bricks capable of much higher working temperatures.

The basic bricks at present employed in open-hearth furnaces making steel, are mostly magnesite bricks—of either the fired or unfired type. It is stated that the fired bricks are preferable where wider checker openings are required, due to their higher refractoriness under load. A notable feature of all such bricks is their markedly higher bulk density. Since their specific heat is still of the same order as that of fireclay bricks they should be capable of storing more heat even if operating at normal temperatures and even more where the maximum temperature at the

top of the setting is, say,  $100^{\circ}\text{C}$ . higher. Their diffusivity would appear to be of much the same order as that of fireclay bricks.

Successful results have been obtained in preliminary trials of bricks consisting essentially of forsterite, which might well prove adequate for the purpose and incidentally be appreciably cheaper than magnesite bricks, whose present price is many times that of normal quality checker brick.

### (c) LIFE AND CAUSES OF FAILURE

In reviewing checker conditions in American furnaces, Sosman says "the temperature is not sufficiently high to cause much reaction of iron oxide with the checker brick in other than the top courses of checkers, and the chief problem is to prevent clogging of the openings with deposits carried over from the slag pocket". With British furnaces the position is frequently quite otherwise, the temperatures carried being so high and the dust carryover so great that the top 6 to 10 courses may be completely destroyed in a matter of 13 weeks. As a result it is frequently the custom to stop at this "intermediate" life and renew this section completely and also sometimes other parts of the setting. The American practice of cleaning the checkers although an admirable one for American conditions would be completely inapplicable to many British furnaces, the top brick courses being well slagged even after very short periods of operation. In American furnaces it is customary to "blow" the checkers with steam or compressed air about every three weeks, whilst at the end of a campaign use is made of sweeping chains or chemical methods to restore the setting to something approaching its original condition.

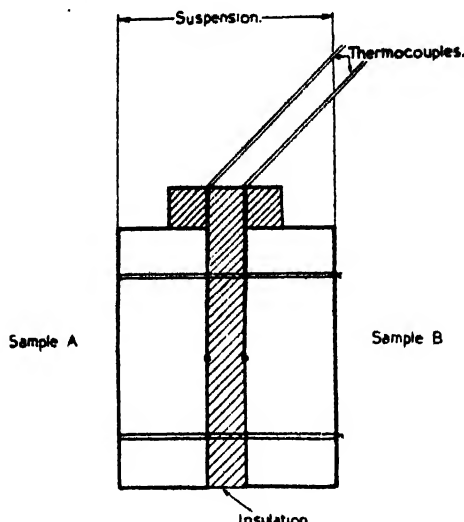
Although the upper courses in British checkers rarely last out the campaign, bricks in the bottom half may well be re-usable. From time to time objection is raised to their being employed again, it being suggested that they are "natureless", *i.e.*, unable to absorb or give out heat in the way that a new brick would. In order to check one such statement, a rather detailed examination was made of certain used bricks, the results of which are given below, since they have an interest extending beyond the particular question as to whether old bricks should be used again. The comparative properties of the used and unused bricks are given in Table XXXVII, from which it will be seen that the discoloured bricks from the gas checkers did show an appreciable pick-up of alkalis at the working face, the melting point of the skin having been reduced to  $1260^{\circ}\text{C}$ . The interior of the brick had a melting point very close to that of the original brick. The crushing strength was well maintained, whilst the bulk density and also the heat

TABLE XXXVII  
USED VERSUS UNUSED CHECKER BRICKS

	Unused brick	Used gas brick		Used air brick	
		Skin	Inside	Skin	Inside
Chemical analysis:	%	%		%	
SiO <sub>2</sub> .. .. .	56.9	53.4	—	55.7	—
Al <sub>2</sub> O <sub>3</sub> .. .. .	33.7	33.4	—	34.0	—
Fe <sub>2</sub> O <sub>3</sub> .. .. .	5.8	5.1	—	5.8	—
TiO <sub>2</sub> .. .. .	0.9	1.1	—	1.1	—
CaO .. .. .	0.1	0.3	—	0.2	—
MgO .. .. .	0.9	0.5	—	0.6	—
Alkalis .. .. .	1.4	5.3	—	1.4	—
ZnO .. .. .	—	0.8	—	0.8	—
MnO .. .. .	—	0.1	—	0.05	—
Loss on ignition, per cent.	0.2	—	—	—	—
Refractoriness, °C.	1560°	1260°	1550°	1260°	1450°
Cold crushing strength, lb. per sq. in. ..	2350	3310	26.4	4960	27.0
Porosity, per cent. .. .. .	26.3	21.7	26.4	21.3	27.0
Bulk density, g.p.ml. .. .. .	1.96	2.04	1.92	2.09	1.93
Average: .. .. .		1.98		2.01	
Thermal conductivity (hot face, 700°C.):					
B.Th.Units .. .. .	6.2	6.2		6.6	
C.G.S. Units .. .. .	0.0021	0.0021		0.0022	
Specific heat (from 750°C.) c.g.s. units ..	0.25	0.25	0.25	0.25	0.25
Heat capacity = specific heat × bulk density	0.49	0.495		0.50	
Diffusivity factor = $\frac{\text{Thermal conductivity}}{\text{Bulk density} \times \text{specific heat}}$	4.3	4.2		4.6	
Permeability to air, c.g.s. units (through both skins) ..	0.0052	0.0071		0.0028	

capacity were higher than those of the original brick, both the conductivity and the diffusivity hardly changed in service. In view of these results the practice of throwing away the bricks from the gas checker because of their suspicious colour was discontinued.

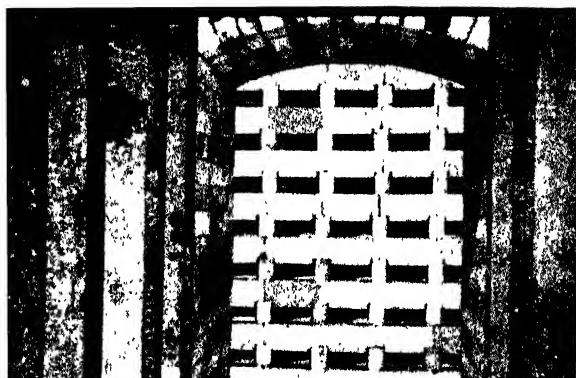
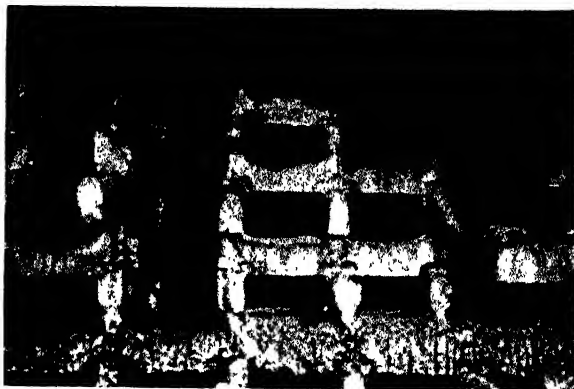
Since the question of heat absorption is a complex one, depending not only on such things as diffusivity, but also such things as surface condition, a trial was arranged, *see* fig. 179, in which 2 different samples,



*Fig. 179. Sandwich method of comparing the rates of heat penetration through two qualities of brick: A and B. Assembly ready for lowering into furnace.*

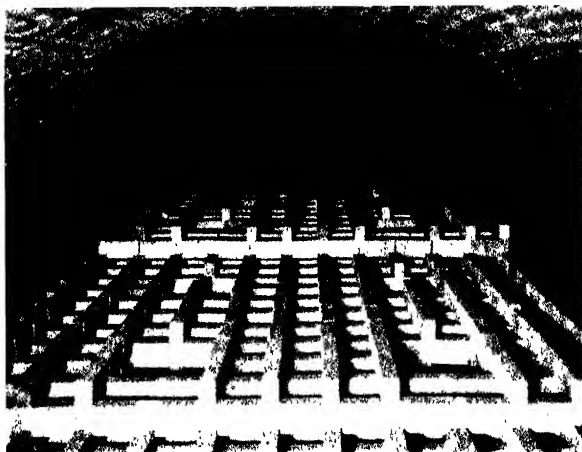
*e.g.*, used and unused, were made into a sandwich having an insulating brick filling. By locating thermocouples at the centre of the faces between the insulating brick and firebrick and lowering the whole assembly into a furnace at  $900^{\circ}\text{C}$ ., the actual rate at which temperature rise occurred as a result of absorption of heat transmission through the test bricks could be compared. The results given in figs. 180 and 181, of two consecutive tests on used and unused air checker bricks, showed negligible differences, whilst similar tests on gas checker bricks also showed no difference unless the used brick was first heated to  $1250^{\circ}\text{C}$ . in order to glaze the surface. The results of this trial were sufficiently encouraging to the operators to try the use of these previously suspect bricks. Any change in efficiency as a result was too small to be observed

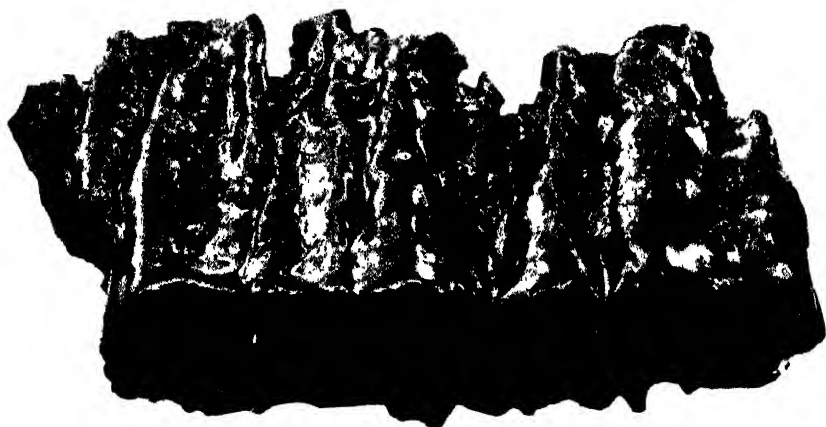
*Fig. 182. Air checker setting (similar to that shown in Fig. 183) after 29 weeks' operation.*



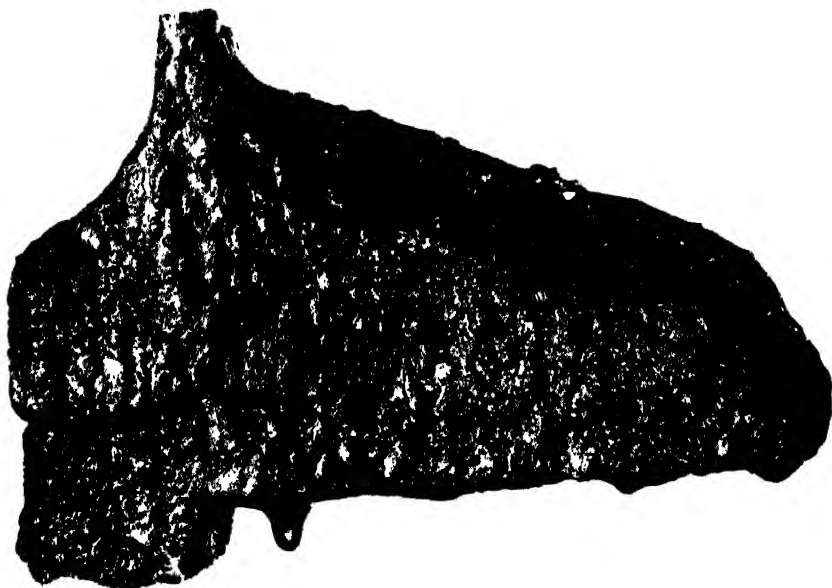
*Fig. 183. New air checker bricks viewed through side wicket.*

*Fig. 184. New air-checker setting viewed from bridge wall.*





*Fig. 185. Semi-silica brick removed from basic open-hearth furnace checker (lowest course visible in fig. 182) after 29 weeks' operation. Note the corrosion of the sides by slag dripping from the top surface.*



*Fig. 186. Silica brick removed from top of basic open-hearth furnace checker (see fig. 182) after 29 weeks' operation.*

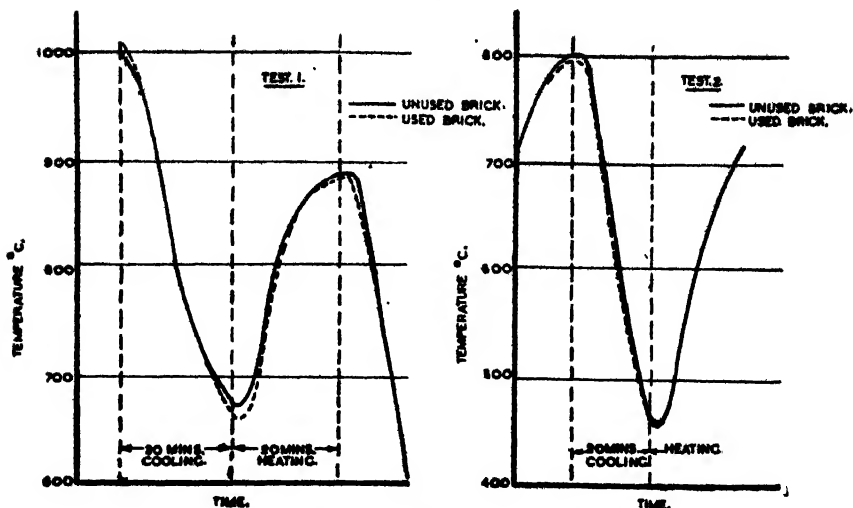


Fig. 180. Heat penetration tests on checker bricks, showing similar rates for used and unused fireclay bricks.

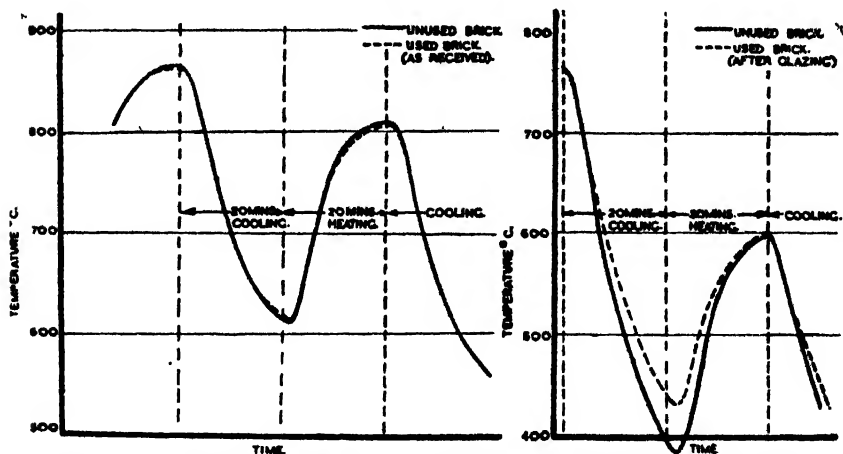


Fig. 181. Heat penetration tests on used and unused checker bricks, showing the lower efficiency of the used brick after glazing.

and the practice of rejecting bricks merely on colour was therefore discontinued.

Those who desire to go into the very complex mineralogy of checker brick corrosion are advised to study the paper by Liang-Ho Su, who carried out a detailed microscopic and X-ray examination of used aluminosilicate checker bricks, and arrived at certain conclusions



regarding the trend of crystallisation of reaction products between slag and bricks. Su agrees that the principal slagging oxides are those of iron and calcium, and suggests that the corrosion process starts with the softening of the glass already present in the bricks, and a consequent increase in the rate of diffusion of the slagging oxides to the interior. He found that oxides such as  $\text{MgO}$ ,  $\text{MnO}$ ,  $\text{ZnO}$ , have a tendency to form spinels, as do also some of the iron oxides. Lime on the other hand tends to form calcium aluminosilicates by reaction with alumina, and calcium metasilicates by reaction with silica bricks.

One difference that must strike any ceramic technologist, is the remarkable difference in the manner of attack with silica, semi-silica and ordinary fireclay bricks. With silica, penetration occurs throughout the brick and the reaction product drips continually on to the courses below, until finally mere skeletons such as that shown in fig. 182, p. 423, remain. Comparison of these with the beautiful setting at the beginning of the campaign (*see* figs. 183, and 184, p. 423) is adequate testimony to the severe conditions existing in small British open-hearth furnace checker chambers. With aluminosilicate bricks there is a tendency for the product to be viscous and to drain down into the courses below, frequently stopping them up. With semi-silica brick (*see* fig. 185, p. 424) the attack is almost completely limited to the working surface, the deposit on the top face gradually wearing the brick away and the reaction product running down the sides to produce the characteristic guttering shown in the photograph. Incidentally the silica brick shown in fig. 186, p. 424, was found to consist almost entirely of large lath-shaped tridymite crystals and glass, a thermal expansion curve not showing the presence of any cristobalite. This presumably indicates that the top checker temperature was normally below the  $1470^{\circ}\text{C}$ . transition temperature.

Table XXXVIII gives the chemical analysis of the skin of a glazed semi-silica brick after use in the gas checker, together with an analysis of the unused brick and of dust samples from the air and gas checkers. These are just the samples taken at random, the actual composition of the corroding material varying not only from one furnace to another, but from one minute to another. The dust analyses being averages of 7 air dust samples and 6 gas dust samples are probably typical of the material carried over in this particular plant. It is, therefore, interesting to note that the only oxides present in consistently high amounts are iron oxide (about 45 per cent.) and lime (about 10 per cent.). Periodically, however, other oxides, *e.g.*, zinc, occur in large amounts (over 20 per cent.), alkalis (over 10 per cent.) and sulphates (over 25 per cent.), their extent doubtless being a direct function of the materials charged.

TABLE XXXVIII  
ANALYSES OF CHECKER BRICKS AND DUSTS

	(1)	(2)	(3) (4)	
<i>Material</i>	<i>Glazed surface of semi-silica brick</i>	<i>Unused semi-silica brick</i>	<i>Checker dusts</i>	
			<i>Air</i>	<i>Gas</i>
	%	%	%	%
Fe <sub>2</sub> O <sub>3</sub> ..	29.4	1.1	48.3	39.3
FeO ..	21.6	—	0.0	0.2
CaO ..	trace	trace	7.9	13.6
MgO ..	trace	0.2	2.6	4.1
MnO ..	trace	0.05	1.8	1.2
SiO <sub>2</sub> ..	39.9	89.6	4.0	17.9
Al <sub>2</sub> O <sub>3</sub> ..	5.9	7.7	3.0	9.9
SO <sub>2</sub> ..	—	—	17.3	6.1
P <sub>2</sub> O <sub>5</sub> ..	—	—	2.6	1.9
ZnO ..	—	—	2.5	0.8
Alkalis ..	1.8	0.6	6.3	1.7
Miscellaneous	0.3	0.5	3.7	3.3

(d) LINES OF IMPROVEMENT

- (1) Since many existing furnaces although greatly increased in bath capacity are frequently still running with the original checker settings, there is little doubt that more ideal arrangements could be evolved by experimentation. Reliable results will, however, only be obtained if trials are carried out under carefully controlled conditions and precise information collected regarding checker top and bottom temperatures and the corresponding preheat levels.
- (2) Where the life of the top courses is at present short, the use of special basic, forsterite or high alumina (55 per cent.) bricks, may well prove economic.
- (3) Where existing fireclay or silica bricks give a satisfactory life, some improvement in thermal efficiency should be obtained from the use of denser refractories.
- (4) Where conditions permit, blowing and cleaning as already generally used in the United States, might advantageously be employed.

## THE CHECKER CHAMBER

(Sections 27-32 and 34-37)

In most British furnaces the checker chamber is entered almost directly from the slag pocket, but in many American furnaces there is an intermediate flue known as the fantail, which appears to result in appreciable drop in temperature of the waste gases. In general the working conditions to which the checker chamber walls and roof are exposed are similar to those found in the checker settings at the corresponding level but other problems arise, notably the need for maintaining gas tightness in a chamber of rather large dimensions.

### (a) CONSTRUCTION

The gas-fired furnace, as developed by Siemens, was equipped with two checker chambers at each end, a small one to preheat the gas and a large one to preheat the air. Even where furnaces have been converted to liquid fuel firing these separate chambers are frequently retained, partly because of the difficulty of maintaining a strong arch over a chamber even greater in volume than that of the air and gas chambers together. More recently this problem has been tackled by departing from sprung arch construction and making use of refinery techniques in which flat suspended roofs are built with specialised types of construction, *e.g.*, those developed by Detrick and Laclede Christy. A further design development now assuming importance is the two-pass checker, in which the gases are first led down through a small high temperature operating unit, then pass up a vertical flue and down again through a second checker chamber operating at a substantially lower temperature. More recently Heuer has suggested that with such construction the filling in the first chamber can be of basic brick, whilst that in the second chamber can be of acid brick. With existing furnaces the installation of such checkers may be prevented by lack of space or excessive pressure drop, but for new installations the arrangement has much to commend it. Where furnaces are fired with producer or mixed gas, the chambers must of necessity be kept separate and it is then desirable to encase them both in steel and to separate the two chambers by a narrow channel that must be kept well ventilated to avoid overheating. In most modern installations both the side walls and chamber arch (whether sprung or suspended) are insulated.

### (b) MATERIALS

For the top sections of the checker chamber side walls and for the checker arches, first quality silica brick or high alumina fireclay brick (42 per cent. or over) should be employed, at least on furnaces having

high driving rates. Where fireclay bricks are used particular attention should be paid to permanent linear change on reheating, since any serious after-contraction may lead to leakage if not to actual collapse of the arch. In the lower sections of the chamber medium alumina fireclay bricks of a quality similar to that used in the setting can be employed. Insulation is normally accomplished by the use of high-temperature insulating bricks in the top part of the checker and low temperature insulating bricks, *e.g.*, diatomite, in the bottom two-thirds, both being built inside a steel casing. In many American furnaces however where the accessibility is great the firebrick construction is not plated, being merely kept tight by external spraying. The charge-side ends of the checker chambers are equipped with wickets which can be knocked out periodically for examination or renewal of the checker bricks. These are generally built of a 9 in. layer of fireclay bricks backed up by insulating brick or sprayed insulation.

#### (c) LIFE AND CAUSES OF FAILURE

With moderate operating rates the life of a checker chamber may vary from 5 to 15 years. False walls are sometimes installed, which can be replaced after attack without interfering with the general structure. Conditions in the chamber are similar to those experienced by the checker bricks themselves, the wear on the walls usually being most severe at the checker top level and in the arch above. The peculiar gouging which occurs is doubtless the result of the waste gas flow pattern and could undoubtedly be reduced by attention to aerodynamic design.

#### (d) LINES OF IMPROVEMENT

- (1) Re-design of the slag pocket and checker chamber arrangement to give more even wear and a better gas distribution through the checker setting. This is an aerodynamic problem, complicated by buoyancy in the checker setting itself. Suspended brickwork should assist in achieving the desired contours.
- (2) Insulation of checker chambers—where not previously carried out.
- (3) Steel plating to give gas tightness and separation of gas and air checker chambers on gas-fired furnaces to reduce the risk of mixing.

### GAS AND AIR VALVES AND FLUES

(Sections 38, 39 and 40)

#### (a) CONSTRUCTION

The design and maintenance of valves is an engineering rather than a refractory problem, but the development of a valve capable of operating

at, say, 1600°C. might have a radical influence on the open-hearth furnace, since it would greatly simplify the design of a one-way fired unit. Maintenance at present temperatures is normally small, but both valves and flues may have a serious effect on maximum firing rate if they are too small and, therefore, produce a substantial pressure drop.

### (b) MATERIALS

Both valves and flues are normally lined with second quality firebrick, though certain firms make use of refractory concrete for this purpose.

### (c) LIFE AND CAUSES OF FAILURE

The life of a flue is normally very long, say 10 to 20 years, mainly because the operating temperatures rarely exceed 800°C. and attack between refractories and corrosive dust brought over from the furnace is, therefore, very slight. The following analyses show that the dust itself would be highly corrosive at high temperatures, due both to its high content not only of iron oxide but of such materials as lead, zinc, and alkalis.

			<i>Flue samples</i>	
			<i>Dense slag</i>	<i>Porous slag</i>
SiO <sub>2</sub>	..	..	45·3 per cent.	42·8 per cent.
FeO	..	..	13·6    ,,	15·2    ,,
Al <sub>2</sub> O <sub>3</sub>	..	..	21·7    ,,	22·0    ,,
MnO	..	..	0·7    ,,	1·6    ,,
CaO	..	..	2·8    ,,	2·4    ,,
MgO	..	..	1·4    ,,	1·4    ,,
SO <sub>3</sub>	..	..	0·1    ,,	0·2    ,,
P <sub>2</sub> O <sub>5</sub>	..	..	0·4    ,,	0·5    ,,
Alkalis	..	..	4·4    ,,	4·0    ,,
CuO	..	..	0·1    ,,	0·1    ,,
SnO <sub>2</sub>	..	..	0·2    ,,	0·1    ,,
PbO	..	..	6·7    ,,	6·4    ,,
ZnO	..	..	2·9    ,,	3·3    ,,

It is sometimes suggested that fireclay bricks for use in gas flues should resist attack by carbon monoxide and consequent carbon deposition. The temperature at which this effect reaches a maximum, *viz.*, 450°C., is frequently obtained, but no damage due to this cause has been observed. More important is the extent to which fireclay bricks in these regions can be replaced by insulating bricks in order, for example, to minimise the temperature drop between a gas producer and a furnace.

## BIBLIOGRAPHY ON THE BASIC OPEN-HEARTH FURNACE (BELOW THE SILL-PLATE LEVEL)

(Exclusive of references included under sections dealing with materials)

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## (a) CONSTRUCTION

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## (b) MATERIALS

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## Chapter X

# THE ACID OPEN-HEARTH FURNACE

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**A**LTHOUGH THIS TYPE OF FURNACE is still responsible for the production of some of the highest quality steel made, *e.g.*, that required for pressure vessels, it is gradually losing its customers to the basic open-hearth and electric steel makers. Its principal claim for retention would appear to lie in the high cleanliness possible by this particular method of steelmaking. The furnace units are generally small in size (10 to 100 tons in capacity) and tend to be designed rather conservatively. Many of the recent furnaces in the United States are oil-fired—unlike their counterparts in Great Britain, which are usually fired with producer-gas.

## FURNACE SECTIONS

The refractories in most sections of acid and basic furnaces are the same, as can be seen from the schedule given as Table XXXIX, the only vital difference being in the hearth, the acid furnace edition of which is built of sintered silica-sand instead of the magnesite or dolomite used in the basic furnace. It is true that fireclay bricks are often used against the pan and that chrome and chrome-magnesite bricks have been tried in various positions, in particular the port ends, the front wall and the downtakes, but it is also true that a satisfactory acid furnace could be built with silica as the sole raw material. In spite of the fact that it contains markedly less of the more expensive materials, the actual refractories' cost per ton of steel made is often greater than that of the basic open-hearth.

## THE ROOF

*(Sections 5 and 6)*

### (a) CONSTRUCTION

The design of roofs for acid furnaces is in no way different from that for basic furnaces. In addition to more conservative designs, Venturi and Maerz types fired with gas or liquid fuel are employed. The same

TABLE XXXIX

## BRICK LAY-OUT FOR ACID OPEN-HEARTH FURNACE

(80-ton furnace—cold pig charge. Water cooled ports and doors.  
Sloping back wall)

<i>Section in diagram</i>	<i>Nomenclature</i>	<i>Material used</i>
1	Gas Ends .. ..	Silica (first quality)
2	Gas Uptakes .. ..	" " "
3	Air Ends .. ..	" " " or chrome magnesite
4	Air Uptakes .. ..	" " " " " "
5	Ramp Roofs .. ..	" " "
6	Main Roof .. ..	" " "
7	Bath Bottom— Sub Hearth .. ..	Silica bricks and medium alumina fireclay bricks
	Monolithic Hearth	Silica Sand
8	Bridge Banks .. ..	Silica (first quality)
9	Banks .. ..	" " "
10A	Gas Port Arch Nose	" (with or without chrome plastic facing), special chrome or chrome magnesite
10B	" " Backing	Silica or ordinary chrome
10C	Wing Walls .. ..	Silica (first quality)
12	Back Wall .. ..	" (fettled with sand)
13	Front Wall .. ..	" (first quality)
14	Doors .. ..	Medium alumina fireclay, second quality silica bricks or rammed chrome plastic
15	Door Arches .. ..	Silica (first quality)
16	Door Jambs .. ..	" " "
17	Gas Slag Arches .. ..	" " "
18	Air Slag Arches .. ..	" " "
19	Gas Slag Walls .. ..	" " "
20	Air Slag Walls .. ..	" " "
21	Slag Wickets .. ..	Used silica bricks
22	Slag Slopes .. ..	Silica (first quality)
23	Slag Bottom Paving .. ..	" " "
24	Slag False Walls .. ..	" (second quality)
25	Slag Top Paving and Protection Walls .. ..	Firebrick and red bricks
26	Checker Bridge Walls	Silica (first quality)
27	Gas Checker Arches .. ..	Silica (first quality) or high alumina fireclay bricks
28	Air Checker Arches .. ..	Silica (first quality) or high alumina fireclay bricks



TABLE XXXIX—*continued*

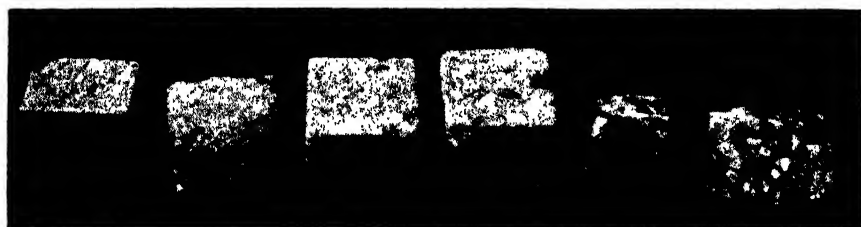
<i>Section in diagram</i>	<i>Nomenclature</i>	<i>Material used</i>
29	Gas Checker Side Walls	Silica (one-third—top) Semi-silica (two-thirds—bottom) H.T. and L.T. Insulation
30	Air Checker Side Walls	Ditto
31	Checker Front Walls	Ditto
32	Checker Wickets ..	{ Semi-silica Used firebricks L.T. insulation
33	Checker Bricks ..	{ <i>Top</i> : Silica (second quality) or high alumina fireclay bricks <i>Bottom</i> : Semi-silica or medium alumina fireclay bricks
34	Checker False Walls ..	Silica (second quality)
35	Checker Bearers and Bearer Walls ..	Medium alumina fireclay bricks
36	Checker Floors ..	" " " "
37	Checker Top Paving	Red brick
38	Gas Valve .. ..	Medium or low alumina fireclay bricks
39	Air Valve .. ..	" " " " " "
40	Flues .. ..	" " " " " "

problems occur in design, for example, the difficulty of making a satisfactory knuckle, and the same precautions must be taken in heating up a new furnace from cold.

#### (b) MATERIALS

The improvements obtained in silica brick quality by careful choice of materials to give low flux content and high bulk density, are equally valuable in the acid furnace, though the proportion of acid furnaces making use of so-called super-duty silica bricks, either for the whole structure or in critical areas, is probably somewhat lower than with basic furnaces. The maximum temperatures obtained are also similar to those experienced in the basic process and there is, therefore, the same necessity for good roof pyrometry.

A property as yet little studied in silica bricks and equally applicable for both acid and basic process is their tendency to glaze. It is generally agreed by melters that a roof that has been "matured" is more capable of withstanding excessive temperature than a new roof, and whether such "maturing" proceeds readily is at least in part a function of grain



*Fig. 187. Glazing test for open-hearth roof bricks. Small cubes heated to  $1690^{\circ}\text{C}$ . for 10 minutes, showing, left to right: no glazing, fluid matrix, even glaze, even glaze, fluid matrix and complete collapse.*



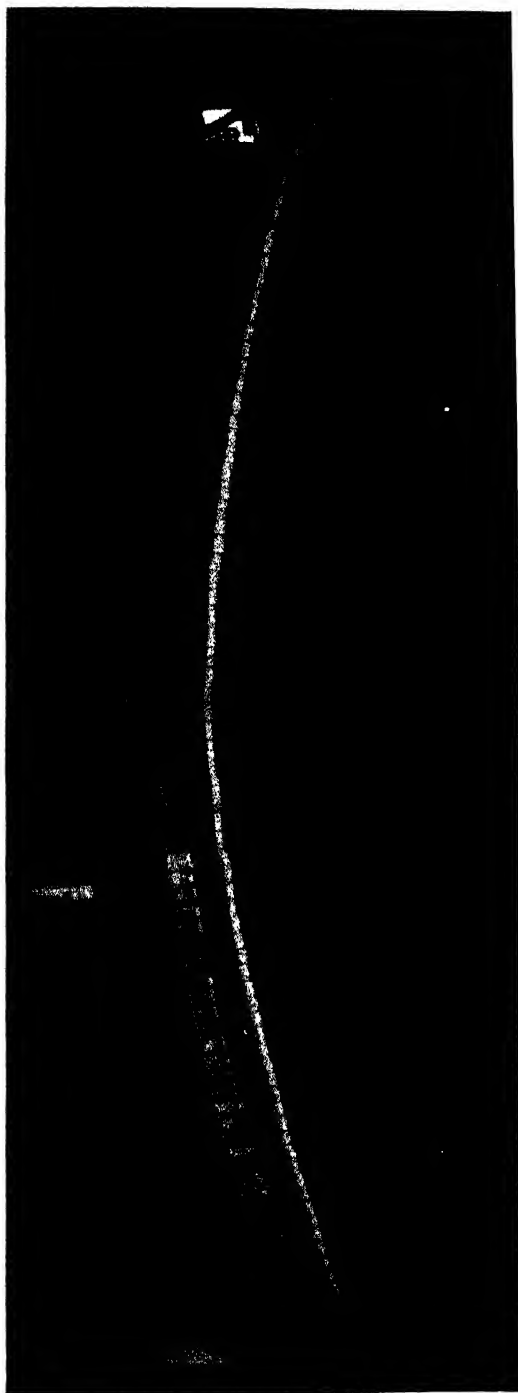
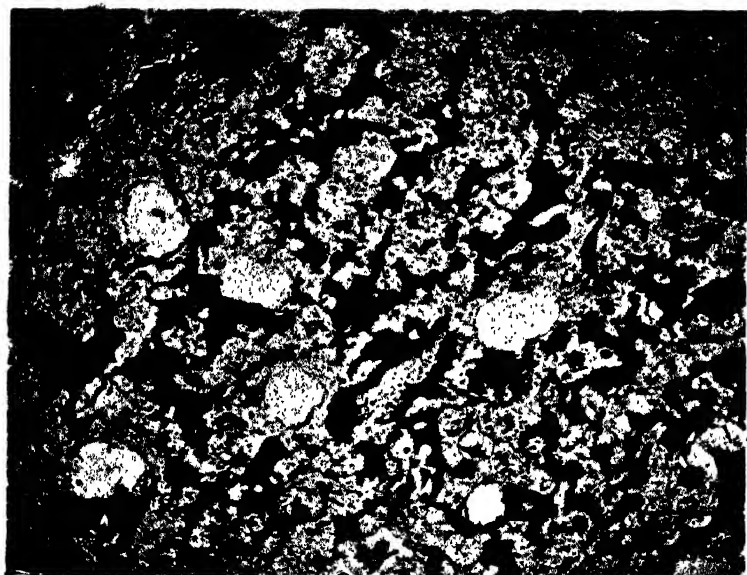


Fig. 188. Excellent condition of acid furnace roof built with silcrete bricks  
(low alumina, high bulk density) after 400 heats.



*Fig. 189. Grey zone of acid open-hearth furnace roof brick, showing magnetite (black) in a cristobalite network. Ordinary light  $\times 40$  (cf. fig. 194).*

and matrix composition. The testpieces, shown in fig. 187, p. 437, which were part of a series intended to elucidate the known service differences between certain markedly different brands of silica brick, indicate a great variation, certain of the test pieces glazing evenly, others showing a fluid matrix and one collapsing completely, although all heated for only 10 minutes to 1690°C. In practice the process of maturing is complicated and undoubtedly assisted by the presence of iron oxide droplets coming from the charge or bath.

### (c) LIFE AND CAUSES OF FAILURE

The lives obtained with acid furnace roofs vary greatly from one plant to another, being as short as 5 weeks or as long as a year. Fig. 188, p. 439 shows a rather remarkable result obtained on one Venturi type furnace fitted with a super-duty (silcrete) roof and fired with producer gas. It will be seen that after over 400 heats the roof was still in excellent condition and that even at the back and front wall comparatively little guttering had occurred. Incidentally the zoned structure and in particular the grey cristobalite face can be clearly seen.

Lives as low as 5 weeks are generally indicative of lack of operational control rather than superb steelmaking. The view was expressed at one time that in order to make pressure vessels, extremely high temperatures and very vigorous bath reactions were necessary, but similar steels giving equally good test results are made today and with furnace lives of nearer 15 weeks. The change has been mainly one of combustion control, including not only measurements and ratioing of gas and air, but also good roof pyrometry and furnace pressure measurement. Extremely long lives on the other hand generally indicate that the furnace is not being driven at what would now be considered the economic rate.

Some 30 years ago, Larsen, Schroeder and co-workers carried out a very intensive study of the conditions existing in acid open-hearth furnaces, whilst even earlier Whiteley and Hallimond made a study which was summarised by Larsen and co-workers as follows:

- (1) The dust in the atmosphere is mainly derived from the metal, both during the melting period and during subsequent boils.
- (2) The furnace atmosphere carries the heaviest burden of solids during the boiling period.
- (3) By weighing checker brick before and after service it was estimated that at least 18 tons of dust was deposited during 120 heats of 70 tons each, or an average of 300 lb. per heat.
- (4) Under a microscope the dust particles from the air checkers appeared as tiny spheres from 1/300 in. downwards in size, often

fritted together to form clusters. The gas checker dust was similar in character, but fritted to a much greater extent.

- (5) The particles are present in the gases as liquid drops. In the melting stages the suspended particles are no doubt due to the spitting of the metal as it fuses, but the origin of the oxide when the molten metal is covered with slag is less certain. It may possibly be due to the presence of iron vapour in the numerous bubbles of carbon monoxide found throughout the boil, or more probably to the spurting of the metal of the bath.

It is salutary to note that it is only now that research workers are beginning to take a serious interest in the nature and concentration of the iron oxide fume referred to by these workers. Quantitative data now being obtained by pushing pure alumina probes through holes in a roof not only shows that the rate of wear is frequently associated with the rate of arrival of iron oxide droplets, but that very big differences occur between one point in the roof and another, and between one part of the heat and another.

The principal difference between the acid and basic process as far as refractories are concerned is that in the former the amount of lime employed per ton of product is only a small fraction of that used in the latter. It is not surprising, therefore, that analyses of zones from roof brick, as shown in Table XL, show no more lime to be present than might be due to migration from the working face into the cooler regions. Comparison of these analyses with those already given for the basic process (Chapter VIII, p. 353) shows that in the latter zones A and B may also contain as much as 5 per cent. lime, most of which has presumably been picked up from the lime and dolomite charged. It will also be noted that as in basic open-hearth furnace roofs, there is a tendency for the alumina to migrate inwards, producing a concentration in what is here described as zone 3.

In Table XLI are given the properties of zones taken from an acid open-hearth roof brick after  $7\frac{1}{2}$  weeks service. It will be seen that there is a marked increase in bulk density in zone B (and a corresponding drop in porosity), due presumably to the absorption of fluxes. What is particularly interesting is the relatively small drop in melting point that occurs in the working face, a figure of  $1680^{\circ}\text{C.}$  being given, compared with  $1700^{\circ}\text{C.}$  for the unused brick. This is due in the main to the relatively small effect which iron oxide has on the melting point of silica, provided other fluxes, such as alumina are present in relatively small amounts and the atmosphere is oxidising. The difference between this melting point and the operating temperature  $1650^{\circ}\text{C.}$  ( $3000^{\circ}\text{F.}$ ) recommended is essentially a safety precaution demanded by the fact

TABLE XL  
ANALYSES OF ZONES FROM ROOF BRICK OF  
ACID OPEN-HEARTH FURNACE  
(After Larsen and Schroeder)

<i>Larsen and Schroeder nomenclature</i> ..	<i>Zone 1</i>	<i>Zone 2</i>	<i>Zone 3</i>	<i>Zone 4</i>	<i>Drips from brick end</i>
<i>Our nomenclature (see fig. 154, p. 357)</i> ..	(A)	(B)	(B <sub>2</sub> )	(C and D)	—
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
SiO <sub>2</sub> .. ..	85.3	72.7	86.4	94.5	79.8
Fe <sub>2</sub> O <sub>3</sub> .. ..	9.0	17.8	3.0	0.3	9.7
FeO .. ..	5.4	6.7	2.3	2.0	6.4
Al <sub>2</sub> O <sub>3</sub> .. ..	0.7	1.2	2.2	1.0	1.4
CaO .. ..	0.1	0.9	6.1	2.4	1.4
MgO .. ..	0.3	0.3	0.4	0.3	0.3
MnO .. ..	0.3	0.4	0.1	0.1	0.7

TABLE XLI  
PROPERTIES OF ZONES IN ACID OPEN-HEARTH FURNACE  
ROOF BRICK  
(80-ton furnace—after 7½ weeks' life)

	<i>Zone A</i>	<i>Zone B</i>	<i>Zone C</i>	<i>Zone D</i>
Length of zone, cm. ..	6.5	6.0	4.5	11.0
Apparent porosity, per cent.	20.7	11.3	21.3	22.5
Bulk density, g.p. ml. ..	1.90	2.07	1.83	1.82
Apparent solid density g.p.ml.	2.38	2.33	2.33	2.33
Permeability to air, c.g.s. units .. ..	0.30	0.043	0.11	0.14
Refractoriness .. ..	1680°C.	1680°C.	1710°C.	1700°C.
Microscopic examination ..	Cristobalite network. Magnetite.	Tridymite —arrow heads. Magnetite and brown glass.	—	—



that certain spots of the roof are liable to be hotter than the point of temperature measurement. It also permits of small errors in temperature measurement, though experience suggests that given tight control the latter need not be more than  $10^{\circ}$  or  $20^{\circ}\text{C}$ .

Microscopic examination (*see* fig. 189, p. 440) shows the cristobalite network in the working face or grey zone. Scattered through this are comparatively large inclusions of magnetite, the whole structure being very similar to that observed with a basic roof. The second zone is also similar, in that it consists mainly of tridymite crystals, often in the form of large arrow-head twins and interspersed with magnetite and brown glass.

#### (d) LINES OF IMPROVEMENT

Apart from designing for more even roof temperature and the strict application of roof pyrometers, the greatest hope for progress in acid roof life lies in the use either of full roofs of super-duty quality or of patches of super-duty bricks in those regions of the roof observed to suffer the most severe wear. In many furnaces, *e.g.*, those of the Venturi type, wear is normally worst in a region adjacent to the back and front wall, but in certain other designs the most rapid deterioration occurs at the roof centre.

It is recommended that where serious local attack is observed, recourse be made to the introduction of alumina probes through holes in the roof, both at the sore point and at other regions not showing the same wear, with a view to finding whether the trouble is due to abnormally high temperatures, excessive flux deposition, or reducing conditions. The flux arrival rate can be measured by determining the amount of iron oxide deposited on a thermocouple sheath in a given time, the thermocouple being used to show the local temperature. The atmosphere can be deduced by using an open-ended tube to take samples at various stages in the heat.

### THE BACK WALLS

#### (Section 12)

##### (a) CONSTRUCTION

As with the basic furnace, the back wall may be either vertical or of the Naismith fully sloping type. As will be shown later, the angle of rest of sand is quite low ( $33^{\circ}$ ), compared with that of fettling dolomite, but the design of back wall does not appear to differ in the two furnaces. In one set of drawings the brickwork, both for the acid and basic back walls, slopes at an angle of  $56^{\circ}$  to the horizontal, while the material

half-way up the wall is shown resting at an angle of about  $48^\circ$ . The point is clearly important, since if the sand tends to run down the wall then a comparatively great thickness will be required at the bottom to ensure that the whole of the wall remains covered. If this thickness is greater than that allowed for, then clearly the designed bath capacity will not be achieved. In practice, however, the fettling layer is thin and the banks sticky, and angles considerably in excess of  $33^\circ$  may be obtained. Most acid open-hearth furnaces do not incorporate sloping back walls, but merely have a vertical silica wall, generally 18 in. in thickness. Where this type of construction is employed care must be taken to allow for adequate expansion, both horizontally and vertically. If the vertical expansion is not adequate the pressure on the roof may shear the bolts on the skew-back channel and cause slipping of the roof during a subsequent repair.

#### (b) MATERIALS

Since back wall life in acid furnaces is normally very short, there is a strong case for using first-class silica bricks and even determining the economics of super-duty qualities. Since the furnace will in any case be heated up at a rate consistent with the use of hard-fired bricks in the roof, the use of equally hard-fired material in the back wall is also desirable in order to minimise trouble due to after-expansion. The use of really soft-fired material (specific gravity 2.48 to 2.52) has been known to lead to excessive shattering of the bricks after only a few days operation. Trials have been made both in Great Britain and the United States of basic back walls, but the general experience would not appear to warrant the use of anything but silica in this position. Chrome-magnesite will normally outlast silica, but the additional life obtained is not sufficient to justify the far higher price, whilst trouble may be experienced due to basic drip cutting the silica banks.

#### (c) LIFE AND CAUSES OF FAILURE

The life of an acid furnace back wall may vary from 3 to 10 or more weeks and can be substantially increased by the use of the fully sloping design. As with basic furnaces considerable damage is doubtless done due to impact of furnace gases (often incompletely burnt) and loaded with iron oxide from the bath. Furthermore, normal design may lead to such attack being more severe than on the front wall, an effect which can be modified, at least in part, by pointing the burner or port slightly towards the front section of the furnace.

(d) LINES OF IMPROVEMENT

In most plants the use of super-duty silica bricks would seem to offer the best chance of a saving, but some improvement might be obtained with sloping back walls by the use of sand graded to give a high angle of rest.

## THE FRONT WALL

(Sections 13, 14, 15 and 16)

The conditions here are similar to those found in the back wall, but the necessity for high quality cement is particularly worth stressing in view of the instability that may otherwise result in the relatively small columns of brickwork. Such an effect was observed in an acid furnace, where the sight-holes permitted a good view of the front wall through the back wall. It was then noticed that shortly after the furnace went into operation, softening of the cement led to its being squeezed out on the inner face, with a consequent tilting of the brick courses which increased with the height up the wall. In the top courses the effect was so marked that bricks actually slid off into the bath. Examination of the cement used showed it to be so high in clay as to be close in composition to the eutectic in the alumina/silica system (1545°C.). Cements used subsequently with complete satisfaction were made either by using a much smaller clay addition to ganister, or by grinding up used roof brick with a small quantity of sulphite lye. Table XLII gives the properties of a number of open-hearth silica cements as tested by Dodd and Green. It will be seen that these all showed over 90 per cent. silica but a refractoriness never under 1660°, except cement *I*, whose analysis was unfortunately not given. It would appear that samples *C* and *F* were bonded with lime and the remainder with a small amount of clay. Closeness of the specific gravity to that of raw quartz suggests that very little crushed silica brick had been used in these particular cements.

Incidentally, cements are frequently used in steelworks with an inadequate check over their suitability for the particular purpose; the mason always being tempted to use the material which will work easily (often because it is high in clay) and very disinclined to use gritty type batches that only retain their water content for a short time after laying. It is, for example, common practice to use two qualities of silica cement in open-hearth furnace construction, one for the above-stage (or hotter section) and the other for the below stage (or cooler section). Since the lower section material—sometimes referred to as “checker cement”—is generally rich in clay and easy to trowel, particular care must be taken to see that it does not get used for the laying of bricks in such positions

TABLE XLII  
OPEN-HEARTH FURNACE SILICA CEMENTS  
(After A. E. Dodd and A. T. Green)

	A	B	C	D	E	F	G	H	I
Chemical analysis (per cent.):									
SiO <sub>2</sub> ..	90.2	90.6	91.0	94.7	92.0	93.6	94.2	—	—
Al <sub>2</sub> O <sub>3</sub> ..	3.5	2.9	1.9	1.8	3.0	2.0	1.0	—	—
Fe <sub>2</sub> O <sub>3</sub> ..	1.4	1.8	0.6	0.6	1.6	0.3	1.7	—	—
TiO <sub>2</sub> ..	0.2	0.3	0.1	0.2	0.2	0.1	0.1	—	—
CaO ..	0.8	0.8	2.6	0.5	0.9	3.5	2.0	—	—
MgO ..	0.4	0.3	0.3	0.3	0.3	0.1	0.6	—	—
Alkalis ..	0.9	0.8	0.5	1.0	0.7	n.d.	n.d.	—	—
Loss on ignition ..	2.1	2.0	2.9	0.6	1.0	n.d.	n.d.	—	—
	99.5	99.5	99.9	99.7	99.7	99.6	99.6	—	—
Screen analysis (per cent.):									
On 20 ..	6.1	16.4	4.0	4.2	10.8	0.4	6.5	3.6	4.1
Through 20 on 30 ..	5.2	6.5	6.2	13.5	8.0	1.0	6.6	13.8	3.7
Through 30 on 60 ..	21.9	14.7	23.3	20.7	26.4	39.5	31.3	43.3	14.2
Through 60 on 90 ..	25.4	12.3	13.1	13.5	22.7	15.1	24.9	12.5	11.3
Through 90 ..	41.5	50.1	53.4	48.1	32.1	44.0	30.7	26.8	66.7
Refractoriness, °C.	1680	1660	1690	1740	1715	1690	1690	1730	1610
Specific gravity ..	2.65	2.65	2.65	2.66	2.59	2.63	2.59	—	—

as front and back walls. That commercial cements do differ greatly in their refractoriness is shown dramatically by fig. 190 opposite, which records the effect of heating a low melting point sand/clay cement and a special silica cement of high refractoriness for 1 hour at 1600°C.

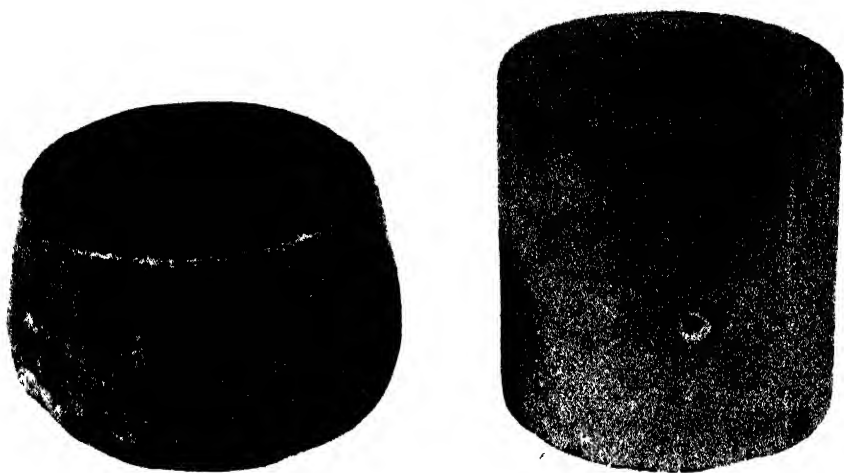
## DOORS

The door problem on the acid furnace is very similar to that on the basic, the usual material being firebrick, but use also being made of water-cooled doors rammed with plastic chrome. Thus Fayles states that doors made in this way have given as many as 150 or even 200 heats. The usual procedure employed is to weld  $\frac{3}{4}$  in. steel studs at 3 in. centres over the door frame and then ram plastic chrome between them. The ramming material is then punched with a steel rod to facilitate removal of water vapour during drying and use. One method of drying suggested is that of passing live steam through the shells of doors connected in series.

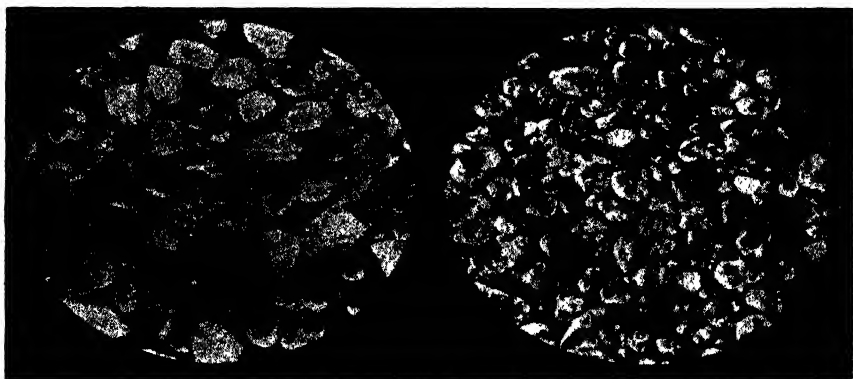
## PORTS

(Sections *roa*, *b*, *c* and *d*)

The gas and air ends (Sections 1 and 3) are normally built of silica brick, though chrome-magnesite has been tried with some success. The port barrel itself in a producer gas furnace is usually built of silica brick, but with fast driven furnaces, *e.g.*, the Venturi type, special chrome or chrome-magnesite bricks are employed, generally in conjunction with water-cooling pipes. The properties of such bricks have already been discussed in Chapter V. When used in conjunction with water-cooling they normally remain reasonably stable for the half-campaign period of, say, 13 weeks, though some deterioration of the nose bricks may occur with possible loss of cooling pipes. The view is sometimes expressed that chrome-magnesite containing basic magnesia may cause damage to the port hearth or end-banks, but in point of fact no such difficulty is experienced, the opposite one of build-up being a greater risk. Acid ports, like basic ports, can be repaired by "Scotch blocking", *i.e.*, the use of a steel former having the shape of the inside of the port on to which ground-up siliceous material (usually gum bonded) is thrown. More recently it has been suggested that such repairs can be done by the use of special materials, *e.g.*, wet ground silica, the cement being dropped through a hole in the port roof. The use of such techniques frequently enables a furnace to be kept in commission that would otherwise have to be taken off, if only because of the risk of damage to the roof from poor flame control.



*Fig. 190. Silica cement test-pieces (A.F.A.) after firing for one hour at 1600°C. Left: Low melting point sand-clay mixture. Right: Special cement of high refractoriness.*



*Fig. 192. Silica sands showing, left: uniform grain size and sub-angular shape; right: non-uniform size and rounded grains. Magnification  $\times 25$  (T. R. Walker).*



*Fig. 193. Influence of grading on the angle of repose. Left: 70-30 sand-silica flour. Right: Straight sand. Cones made by pouring sand through funnel.*

As with front and back walls, the quality of the jointing cement used with port bricks is of the greatest importance. If the cement is not of similar quality to the bricks then it may well disappear prematurely, allowing gas to leak through on the incoming end with damage to the port and an unfavourable effect on the furnace as a whole. The use of special cements in this position, *e.g.*, that illustrated in fig. 190, p. 449, may well increase port life by as much as 50 per cent.

## THE HEARTH

### (a) CONSTRUCTION

The thickness of brickwork in the sub-hearth of the acid furnace is very similar to that used in basic furnaces, though the materials are different. There are usually two courses of medium alumina fireclay brick next to the pan on which are laid three courses or so of first quality silica brick, making a total brickwork thickness of 15 in. Expansion allowance should always be left but the precise procedure employed varies considerably from one plant to another. In some plants gaps as big as one inch are left within the furnace chamber, whilst in others the expansion allowance is all under the banks, where the risk of fouling by debris is much reduced. That some expansion allowance is required was shown by one trial in which the joints became blocked and the whole of the silica sub-hearth lifted when the furnace was heated up. Fayles, being concerned with front wall breakouts, suggests that the side wall opposite the top of the hearth should contain a chrome-magnesite brick section. To avoid any difficulties with the burning of sand on to the chrome brick he builds the inner face in silica.

The monolithic section of the hearth is almost always formed by fritting sand on to the brick sub-hearth layer by layer, each one being fused to a glass before the second is applied. Nicholas refers to trials made with sand bottoms rammed with molasses as a bond, but the use of such hearths does not appear at all widespread. Since with the normal port position it is difficult to obtain high temperatures on the top of the sub-hearth, recourse is sometimes had to the letting down of the sand with less pure material. Thus Ferguson refers to a brown sand containing 11 per cent. alumina,  $2\frac{1}{2}$  per cent. iron and 2 per cent. lime, which is mixed to the extent of 25 per cent. in the bottom layers, the percentage present being gradually reduced as the top is approached. Where oil firing is in use no such difficulty need be experienced, since it should be possible to tilt the burners downwards during the early stages of hearth making. After the hearth has been burned in, which normally takes several days, a slag melt is carried out, so that any cracks or holes are filled with slag rather than molten metal.



**(b) MATERIALS**

The fireclay and silica bricks used in hearths are normal qualities, but the properties of the sand are to a large extent peculiar to this particular use. Any generalisation is extremely difficult, since the sand employed is very much a matter of local availability. Thus, prior to the Second World War, Belgian sand was fairly standard in Great Britain, but during the war perfectly satisfactory bottoms were built and maintained with material mined in Great Britain. What is clear is that the content of certain impurities, notably alumina and alkalis, should be low, since otherwise the flux content at working temperatures will be high and the hearth will be soft. The properties of a number of sands used successfully are summarised in Table XLIII. Although these particular tests were carried out some time ago, more recent data suggests that there has been no fundamental change in the properties. The precise screen analyses and impurity contents vary not only from plant to plant but from consignment to consignment.

Considerable attention has recently been paid in the United States to the properties of acid open-hearth sands and those desiring an impression of American practice would do well to study the numerous discussions in the A.I.M.E. Open-Hearth Proceedings, *e.g.*, those following the paper by Kopec in the 1953 volume, where such matters as screen analysis, chemical analysis, clay content, crystal shape and refractoriness, are discussed. In this same series of papers an attempt is also made to consider statistically the relation between such factors as sand consumption and hearth area.

**CHEMICAL ANALYSIS**

It will be seen that all the sands in Table XLIII have a silica content of over 98 per cent. and with one exception an alumina content of less than 0.5 per cent. The necessity for such clay free sands is clearly seen from a study of the alumina-silica equilibrium diagram (fig. 117, p. 247). Even a small amount of alumina (or alkalis) being sufficient to drop the melting point of silica below steelmaking temperatures. The iron oxide content of such sands is normally low, though this is not as vital, since both iron and manganese are soon picked up from the bath.

**GRADING**

The sands normally employed for fettling acid open-hearth furnaces have such a limited grain size range that a large number of sieves are required to cover the significant fractions of the grading. The typical range is shown in Table XLIII, together with the equivalent sieve

TABLE XLIII  
PROPERTIES OF ACID OPEN-HEARTH FURNACE SANDS

	Belgian		British			
	I		I	2	3	4
Chemical analysis:			per cent.	per cent.	per cent.	per cent.
SiO <sub>2</sub> .. .. .	99.47	..	99.07	99.20	98.38	99.67
Al <sub>2</sub> O <sub>3</sub> .. .. .	0.25	..	0.47	0.34	0.84	0.08
Fe <sub>2</sub> O <sub>3</sub> .. .. .	0.03	..	0.11	0.11	0.10	0.06
TiO <sub>2</sub> .. .. .	0.02	..	0.03	0.05	0.03	trace
CaO .. .. .	trace	..	trace	trace	trace	trace
MgO .. .. .	trace	..	trace	trace	trace	trace
Alkalis as K <sub>2</sub> O ..	nil	..	nil	nil	0.47	0.14
Loss on ignition ..	0.22	..	0.32	0.30	0.18	0.14
Grading						
B.S.I. sieves		Sieve aperture				
(per cent. remaining on each sieve)		mm.				
On 5 mesh .. ..	..	3.4	..	..	..	..
On 10 " .. ..	..	1.7	0.5	..	..	..
On 16 " .. ..	..	1.0	1.0	0.5	..	1.0
On 22 " .. ..	..	0.70	6.0	0.5	..	8.0
On 30 " .. ..	..	0.50	19.0	5.0	4.5	41.0
On 44 " .. ..	..	0.35	41.4	13.2	37.1	34.0
On 60 " .. ..	..	0.25	21.1	39.8	36.4	13.5
On 72 " .. ..	..	0.21	5.0	15.0	9.5	1.0
On 100 " .. ..	..	0.15	4.0	18.0	10.0	1.0
Through 100 mesh	..	< 0.15	2.0	8.0	2.5	0.5
Microscopic examination ..	..	..	Moderately rounded	Sub-angular	Sub-angular	Very rounded
Angle of rest (cone method)	..	..	32°	31°	31°	30°

apertures in mm. It will be seen that 82 per cent. of the Belgian sand passes through a 44-mesh sieve and remains on a 72-mesh sieve. The limited range of particle size of this material as compared with that of a typical British sand and the specification for one German sand are brought out in fig. 191. It will be seen that the British sand covers a

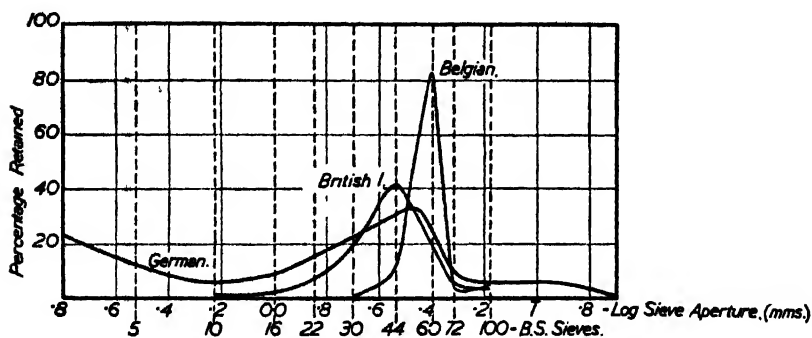


Fig. 191. Grain size distribution for British, Belgian and German open-hearth furnace sands.

wider range than the Belgian, while the German material would be better described as a crushed sandstone, since it contains 23 per cent. of over 6.5 mm. material. According to Abker, even coarser sands are employed in Germany, but the use of material of over 1 cm. in diameter is not recommended, since it is stated that it may "spall" when heated with such violence as to cause injury to the operators. Ferguson suggests that the white sand "must consist of clear coloured, uniformly sized, rounded crystals. It should not contain angular, irregularly shaped masses and fines which will not build into uniform layers on the furnace banks". This is an accurate description of Belgian sand, which has hitherto been considered a standard of excellence, but as will be shown in a later section on lines of improvement, it does not necessarily represent the ideal acid furnace hearth material.

### Micro-structure

Examination of sands under the microscope provides information regarding the shape and the nature and distribution of the impurities. Characteristic photographs given by T. R. Walker in his book on Foundry Sands are shown in fig. 192, p. 450. There would not appear to be any definite evidence as to whether a rounded grain is superior to an angular one, either for bottom making or for fettling.

### *Refractoriness*

First quality open-hearth sands do not melt at all readily until heated to about  $1750^{\circ}\text{C}.$ , *i.e.*, a temperature well in excess of the melting point shown for silica in equilibria diagrams. They doubtless "melt" at appreciably lower temperatures, but the glass formed is so viscous that higher temperatures are required to produce appreciable flow. Such temperatures are not readily attained with ordinary ceramic equipment, but if a sillimanite crucible be filled with such sand and heated fairly rapidly to this temperature, it will be found that the top layers have gone to a clear glass. Other sands, such as No.3 in Table XLIII, will yield a glass at  $20^{\circ}$  or  $30^{\circ}\text{C}.$  lower temperature. Since there is a very small margin of safety between the tapping temperature of the steel and the refractoriness even of clean sand, and since this refractoriness must be brought down considerably by the pick-up of impurities, the advantage of using the most refractory sands available should not require emphasis.

### *Angle of repose*

If sand is poured through a funnel on to a bench it assumes a cone shape and any attempt to build it up above a certain angle does not succeed, provided the material is really dry (*see* fig. 193, p. 450). The angle which the surface of the cone makes with the horizontal, which is known as the angle of rest or repose, is comparatively small ( $30^{\circ}$ – $33^{\circ}$ ), but, as will be seen later, can be made to vary considerably by artificial means, such as the addition of a more finely ground fraction.

### *Sintering tests*

The relative ability with which different sands are likely to sinter in practice can be determined by ramming them into 1 in. holes in silica brick and firing to  $1650^{\circ}\text{C}.$ , say for half an hour. Under these conditions some pure sands (particularly when coarse) show relatively little signs of sintering, whilst other sands, of lower grain size, and substantially higher alumina content, sinter quite strongly.

### (c) LIFE AND CAUSES OF FAILURE

As with basic furnaces, the actual life obtained on a bottom is very much a matter of local practice, since bottoms can be "flowed" and built up again, or repaired locally, almost indefinitely. There is no doubt, however, that the older the bottom the greater the chance of a breakout and the higher the heat loss to the pan. At the 1941 American Open Hearth Conference, it was suggested that a life of 1000 heats could

be considered satisfactory, but bottoms many times this age are still in use. The "flowing" procedure is carried out by throwing lime on to the hearth when it is at a very high temperature and allowing the slag so formed to drain off through the taphole. When perhaps one-third to one-half has been run away the original contour is recovered by sanding and sintering as with a new hearth.

The changes undergone by a hearth in service were illustrated in a classical paper by Whiteley and Hallimond, who showed, from samples taken on used hearths, that the main change was that of iron oxide absorption and conversion of the silica in the working face to cristobalite. Table XLIV shows a set of hearth analyses given by

TABLE XLIV  
COMPOSITION OF THE WORKING HEARTH  
(60-TON ACID FURNACE)  
(After J. M. Ferguson)

<i>Analysis (per cent.)</i>	<i>1</i>	<i>2</i>	<i>3</i>	<i>4</i>
SiO <sub>2</sub> .. .. .	70.54	69.24	67.4	73.6
FeO .. .. .	15.84	18.00	20.4	17.5
Fe <sub>2</sub> O <sub>3</sub> .. .. .	5.51	4.44	3.96	3.44
Al <sub>2</sub> O <sub>3</sub> .. .. .	2.29	2.65	1.06	2.06
MnO .. .. .	5.28	4.05	5.02	2.27
CaO .. .. .	0.08	1.04	2.10	0.84
MgO .. .. .	0.72	0.86	0.44	0.21
	100.26	100.28	100.38	99.92
Bottom in service ..	12 weeks	13 weeks	8 years	8 years

Ferguson, from which it will be seen that the analysis on an eight-year-old hearth is much the same as that of one in operation for only 12 weeks; both contain over 20 per cent. iron oxide and a ratio of FeO to Fe<sub>2</sub>O<sub>3</sub> considerably greater than that for magnetite. The alumina content is only of the order of 2 per cent., while the manganous oxide runs at 4 per cent. and the lime at 1 to 2 per cent. There is little doubt that surface layers of this composition form a silica network, the pores of which are filled with relatively fluid material—any further absorption of flux leading to the solution of the working face and the necessity, therefore, to fettle.

As with the basic hearth, considerable damage is done by metal left on the hearth after tapping. By oxidising it provides a local excess of iron oxide and, therefore, also a localised softening of the bottom. To minimise this effect metal pockets should be rabbled out as thoroughly as possible and dried up with silica sand. Such difficulties can be in part avoided if sufficient attention is paid to the maintenance of an adequate slope to the taphole from all points on the hearth.

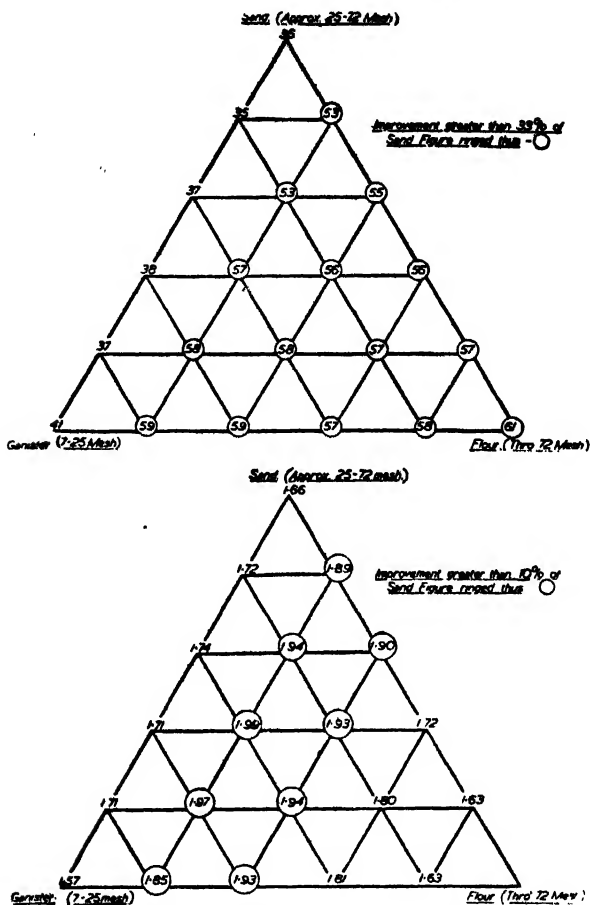
The similarity of the working face of a hearth to a grey zone in the roof, is clearly brought out by figs. 189, p. 440, and 194, p. 459, which both show a continuous network of cristobalite with magnetite and other impurities in the interstices. Lower in the hearth and higher in the roof the cristobalite changes in accordance with equilibrium expectations to tridymite (fig. 195, p. 459), while still lower in the hearth or away from the hot face of the roof residual quartz can be found.

The amount of silica sand used in making the original hearth is only a small part of that used for fettling, a fact that should be borne in mind when bottom trouble is experienced and the desirability of a completely new bottom is discussed. The actual fettling consumption varies greatly from plant to plant, figures as different as 45 and 120 lb. per ton of steel being quoted. This means that on, say, an 80-ton furnace anything from 2 to 4 tons of sand dissolves during a heat and have to be replaced by subsequent fettling. If, therefore, the renewal of the hearth results in any appreciable drop in fettling time and consumption, it may well prove in the long run to be an economy. This will be even truer when satisfactory methods of ramming hearths replace the present rather laborious procedure of burning-in—a change which has already occurred in the basic field.

#### (d) LINES OF IMPROVEMENT

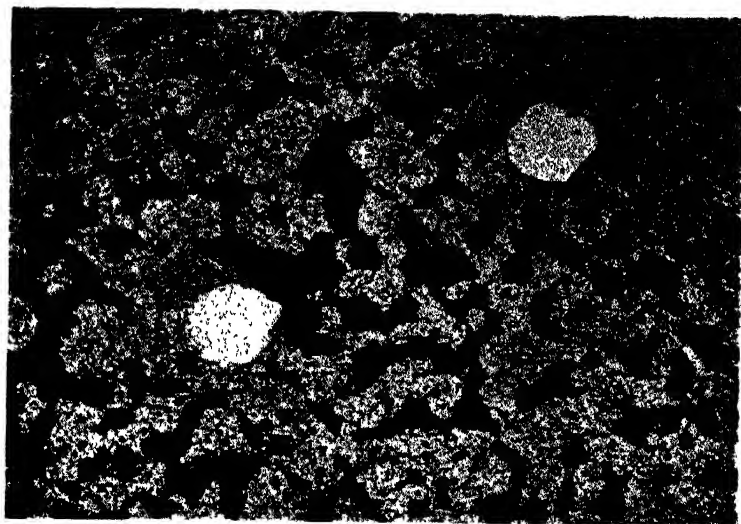
If a pint jar is “filled” with marbles there is still room in it for about half a pint of water. In other words such a packing has an intergranular porosity of roughly 50 per cent. If rounded sand grains were used instead of marbles it would be found that the resulting porosity was of the same order. Open-hearth furnace sand is not strictly uniform. It has a limited size range, which tends to increase its packing density. In actual practice the porosity obtained will be well under 30 per cent. because of the softening and slagging of the mass at temperature. Even so there is no doubt that considerable improvement in slag resistance would be obtained from a graded silica batch. The objections to the use of a range of sizes, including, say, silica flour, are mainly that such mixtures tend to segregate on throwing and that unless special precautions are taken

there is likely to be a silicosis risk when finer sizes are employed. That a marked improvement both in packing density and in angle of rest can be obtained by graded batches is clearly brought out by figs. 196 and

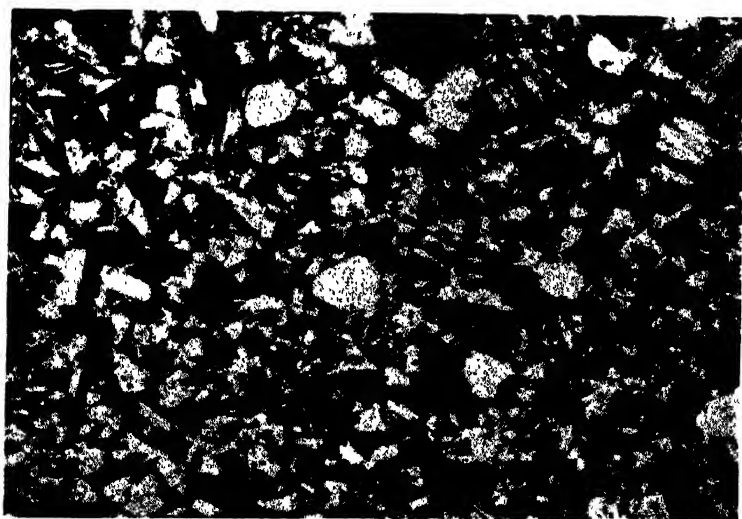


Figs. 196 and 197. Packing density (below) and angle of repose (above) of ganister (coarse)—sand (medium)—silica flour (fine) mixtures.

197, and Table XLV. It will be seen, for example, that the packing density of certain mixtures of ganister, sand and silica flour, was as high as 1.99 compared with only 1.66 for sand alone. Further that such a mixture has an angle of rest of 57° compared with 36° for sand. The packing density figures were got by the use of an A.F.A. rammer (see fig. 198, p. 460) and might reasonably be expected in practice were

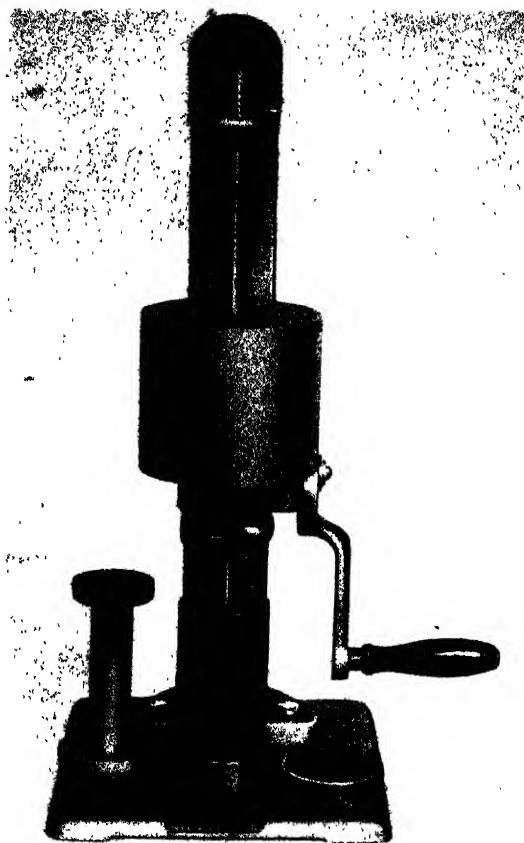


*Fig. 194. Grey zone of acid open-hearth furnace bottom, showing magnetite in cristobalite network (cf. fig. 189). Ordinary light  $\times 40$ .*

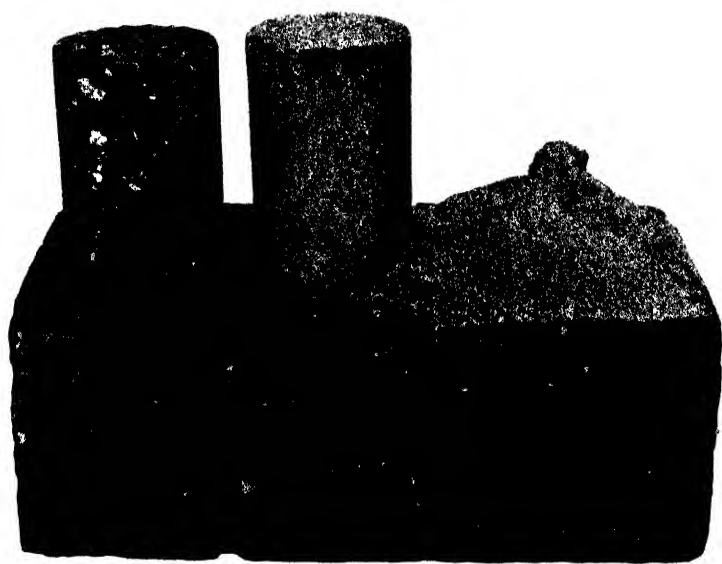


*Fig. 195. Black zone behind the working face of open-hearth furnace bottom showing tridymite laths. Ordinary light  $\times 40$ .*

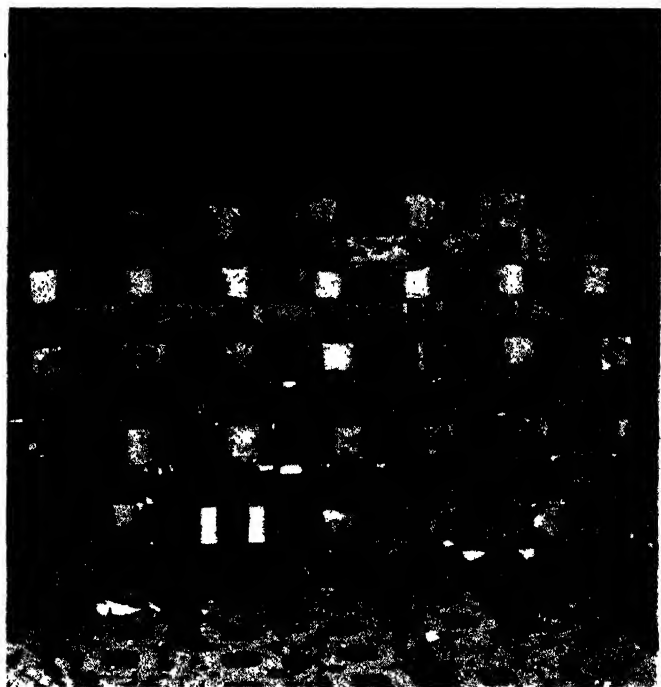




*Fig. 198. A.F.A. sand rammer  
used for packing density tests  
(Ridsdale & Co. Ltd.).*



*Fig. 199. Sinter test-pieces after one hour at 1600°C. Left to right: Ganister-sand-silica flour, sand-silica flour, and straight sand.*



*Fig. 200. Acid open-hearth furnace checkers showing excessive choking with iron oxide after only 7½ weeks' operation.*

TABLE XLV

PACKING DENSITY AND ANGLE OF REST OF  
GANISTER-SAND-SILICA-FLOUR MIXTURES

Batch Number	Mixture (B.S.I. sieves)			Packing density	Angle of rest
	Ganister (7-25 m.)	Sand (25-72 m.)	Silica flour (Through 72 m.)		
	per cent.	per cent.	per cent.	g.p. ml.	
1	80	20	0	1.71	36° 41'
2	0	20	80	1.63	57° 28'
3	40	20	40	1.94	57° 48'
4	20	0	80	1.85	58° 54'
5	0	80	20	1.89	52° 59'
6	40	40	20	1.99	56° 57'
7	20	80	0	1.72	35° 11'
8	20	0	80	1.63	58° 27'
9	20	40	40	1.93	55° 44'
10	60	40	0	1.71	37° 46'
11	60	0	40	1.93	58° 45'
12	60	20	20	1.97	57° 48'
13	40	60	0	1.74	37° 0'
14	0	60	40	1.90	54° 59'
15	20	60	20	1.94	53° 12'
16	0	40	60	1.72	56° 7'
17	40	0	60	1.81	57° 28'
18	20	20	60	1.80	56° 48'
A	100	—	—	1.57	40° 30'
B	—	100	—	1.66	35° 54'
C	—	—	100	n.d.*	60° 32'

\* Flowed around the plunger.

such batches rammed in layers on the sub-hearth. Experiments along these lines and using a tar bond have been carried out in the works, but considerable further development would be necessary before they became practicable. One serious problem is that with such batches there is a marked expansion on firing, due to the conversion of quartz to cristobalite, which may well shatter the hearth. In an enclosed position, however, such as a taphole, no difficulty due to shattering need be expected. Experiments have also been done with moist sand flour mixtures for fettling. Here again the preliminary trials were encouraging, but no general change in practice has yet occurred.

The angle of rest figures, which are of interest in relation to the fettling of banks, were obtained by piling the mixtures on a shelf 15 in. long and 7 in. wide. The angle of rest obtained by measuring the height and width of the columns are some 3° higher than that given by a poured cone, but are comparable within themselves and more reproducible than the results on cone tests. That such mixtures sinter more readily than straight silica sand is clearly demonstrated by the condition of the A.F.A. test pieces shown in fig. 199, p. 461, which were fired together for one hour at 1600°C.

## GAS AND AIR UPTAKES

(Sections 2 and 4)

In most acid furnaces the uptakes are still built of silica bricks and tend to wear out fairly rapidly due to the very high iron oxide pick-up. More recently certain firms have adopted the use of chrome-magnesite in this position, partly in order to increase downtake life, but also because the use of basic bricks in this position reduces the risk of "curtains" forming across the fantail openings. Such curtains can easily reduce furnace draught and slow up production to a point where a furnace has to be taken off. The mechanism of attack on silica bricks in this region has been described by Larsen and Schroeder, who find the conditions to be similar to those existing in the roof except that the concentration of iron oxide in the working face tends to be still higher, *viz.*, about 20 per cent.

Both the gas and air slag pockets (Sections 17-25) and the lander are similar to those employed in the basic process and will not, therefore, be described here.

## THE CHECKERS

(Section 33)

There is little difference in practice between acid and basic checkers, though some firms find that their acid checkers fail by choking (*see* fig. 200, p. 462) rather than erosion. This is, however, in part a matter of furnace design and driving rate. Even on basic furnaces variations in checker top temperatures from 1200 to 1500°C. are quite common. The amount of slagging expected for similarly severe operating conditions would, however, be slightly less in view of the absence of lime and dolomite dust and the relative purity of the scrap and pig iron charged to the acid furnace. The mechanism by which iron oxide is carried over to the checker chambers is still something of a mystery. Until recently it

was assumed to be due to volatilisation of  $\text{FeO}$ , but more recent studies on basic furnaces show that maximum fuming does not coincide with maximum furnace temperature—the worst fuming often occurring  $100^{\circ}\text{C}$ . before the top roof temperature is reached and, being also greatly affected by the type of fuel used and the amount of fluid used for atomisation. Thus the use of pitch-cresote tends to result in more fuming than occurs with oil, whilst high steam/oil ratios tends to give less fuming than occurs with low steam/oil ratios. Such evidence as is available supports a German suggestion that an unstable compound of iron, carbon and oxygen, for example a carbonyl, is formed at the surface of the scrap and subsequently dissociates to give metallic iron which then oxidises to give an extremely finely divided product. Coarse droplets of iron oxide are probably in the main eliminated by the right angle turn into the downtake and again in the slag pocket. The much finer fume particles are partly trapped in the checkers and partly pass up the stack.

The construction of the checker chambers (Sections 27–32 and 34–37) and also the gas and air valves and flues (Sections 38, 39 and 40) is similar to that on a basic furnace.

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## *Chapter XI*

# ACID AND BASIC BESSEMER CONVERTERS

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IT IS JUST OVER one hundred years (1856) since Henry Bessemer presented his paper to the British Association on "The Manufacture of Malleable Iron and Steel Without Fuel". Purists may quibble at the title of his lecture, since it can be argued that the metalloids in the molten iron are the fuel in this process, but no one can doubt either the courage that led him to make these experiments or the success achieved. By 1889-90 acid bessemer steel reached a record figure in Great Britain of 1,750,000 tons per annum, completely dwarfing the amount available by previously conceived processes and representing some 48 per cent. of the British steel output at that time. Other types of furnace have since outpaced the bessemer, partly because relatively little iron ore and, therefore, iron, of a sufficiently low phosphorus content is available in Great Britain; this element not unfortunately being removed in the acid bessemer process.

Another major step forward occurred in 1876, when Sidney Gilchrist Thomas showed the possibility of removing phosphorus when using a basic lined converter. This method was adopted more generally on the Continent than in Great Britain, a substantial part of Continental steel still being made in this way.

The chief competitor to both the acid and basic bessemer processes is, of course, the open-hearth furnace, which although invented somewhat later is now used for the bulk of the world's steel production. The main reasons for its adoption are to be found in the higher metallic yield, the greater degree of control possible, and consequently the ease with which special quality steels can be produced.

Quite recently, and as though in celebration of its centenary, the bessemer process has taken on another new lease of life. In addition to the increased use of acid side-blown converters for the production of steel castings, new processes, for example, the "L-D", and the rotor, have been developed. In the "L-D" process, first operated at Linz and Donawitz, Austria, batches of about 30 tons of molten pig iron are charged into a vessel similar in shape to the bessemer converter but without tuyeres in the bottom or the side. Instead of an air blast passing



through the metal, or blowing tangentially on to it, a jet of relatively pure oxygen is blown vertically downwards on to the top surface. The pressures employed are far higher than in normal bessemer procedure (over 100 lb. per sq. in.) and the oxygen, which is supplied at the rate of about 3000 cu. ft. per minute, is so effective that the 30-ton charge can be converted to steel in about 20 minutes. The obvious problem, namely the provision of a suitable tuyere, through which to introduce the gas, has been solved by the use of a water-cooled copper lance. Introduced through the throat this projects downwards to within a few feet of the metal surface. One of the main advantages of this new process is that basic bessemer steel can be made with a low nitrogen content. This, together with the speed and efficiency of production, are likely to lead to its increased use at least in regions where relatively low phosphorus iron are available.

This development, which has caused so much interest in recent years, is by no means the only revival on the bessemer front, great interest also being shown in the oxygen-steam process, as operated, for example, by Haspe, and the oxygen blown rotor processes under development in Germany and Sweden.

The refractories technologist studying the bessemer converter for the first time, must be immediately struck with two features of the process, *viz.*, the very large outputs obtained with a given size of plant and the extraordinarily rapid rate of wear observed on the refractories. Even a medium size converter can turn 25 tons of iron into steel in about 12 minutes, whereas an open-hearth furnace, occupying a considerably greater ground space, takes nearer 12 hours to complete a heat.

The following comparison, although rather unorthodox in concept, at least serves to show that on a hearth area basis the relative outputs of the two processes are roughly 100 : 1.

			<i>Acid bessemer converter</i>	<i>Open-hearth furnace</i>
Capacity	..	..	25 ton	80 ton
Conversion time	..	..	12 min.	12 hr.
Hearth area	..	..	60 sq. ft.	350 sq. ft.
Production rate—				
tons per sq. ft. of hearth area				
per hour	..	..	2.08	0.02
lb. per sq. ft. hearth area				
per hour	..	..	4660	42.5

As will be seen later it is this rapid production that offsets the relatively rapid wear on the refractories and results in a refractories cost substantially lower than that obtained with the open-hearth process.

The actual size of converters (*see* figs. 201 and 202, p. 471; and 203, p. 472) varies widely but a capacity of 25 tons may be considered fairly typical both of the acid and basic process. It should be noted, however, that, particularly in Germany, basic converters of 45 tons capacity are often employed. Side blown converters on the other hand tend to be much smaller, generally of the order of 5 tons capacity. They also differ from the other converters in that they normally take cupola metal rather than iron direct from the blast furnace.

It may well be asked why the bessemer process has not overtaken the open-hearth in view of the great advantage of rapid production from a small plant and the low refractories cost. The answer, apart from the availability of suitable grades of hot metal, appears to lie mainly in yield, which is normally considerably lower than with the open-hearth and on specifications which frequently do not permit the use of bessemer steel, often because of alleged brittleness due to high nitrogen content. If this latter difficulty is overcome, and in addition the use of iron of higher phosphorus content becomes practicable through the rotor process then the proportion of bessemer steel may once again tend to increase. The amounts of steel made by the different processes in Great Britain during 1955 are summarised below:

	<i>Millions of</i>				
	<i>tons</i>				
Open-hearth—acid..	..	..	..	..	1·00
„ „ —basic	..	..	..	..	16·25
Bessemer—acid ..	..	..	..	..	0·25
„ —basic ..	..	..	..	..	1·03
Electric—arc ..	..	..	..	..	0·97
„ —H.F. ..	..	..	..	..	0·13
Miscellaneous ..	..	..	..	..	0·16
Total ..	..	..	..	..	19·79

Proportions in other countries differs substantially from these, particularly on the Continent, where the basic bessemer process supplies a substantial part of the total output.

With both processes the molten iron used is normally taken from a mixer rather than direct from the blast furnaces. This vessel serves a dual purpose, on the one hand acting as a surge vessel thus ensuring that metal is always available, and on the other hand helping to even out the analysis of the iron and, therefore, also of the product. Some scrap is also used, though generally not more than 10 per cent. This is introduced towards the end of the blow in the acid process but in the basic

process it is put in soon after the blow commences. Incidentally an attractive feature of the new processes employing oxygen is the possibility of using a higher proportion of locally arising scrap.

## INACTIVE MIXERS

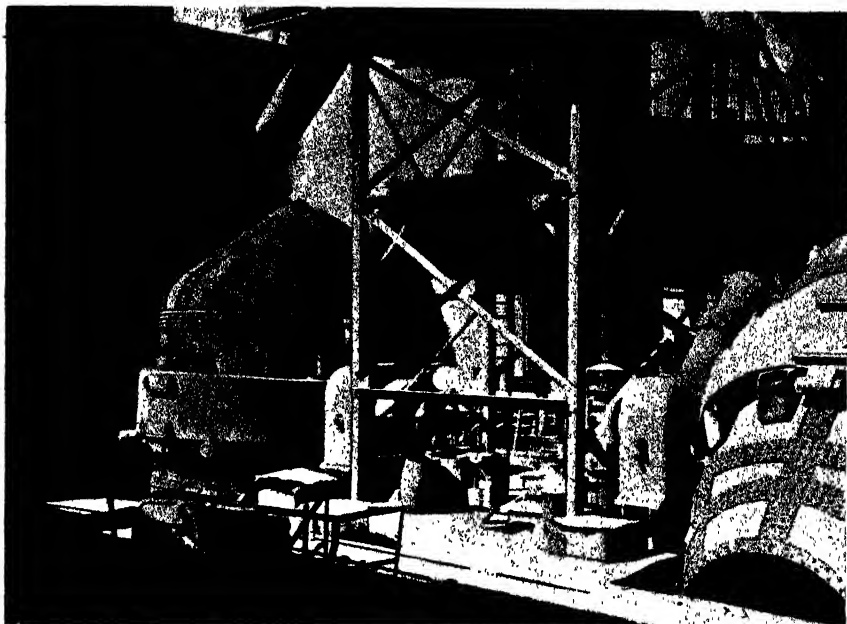
The term inactive mixer is used to distinguish this cylindrical drum type container from active mixers which are essentially large open-hearth furnaces having a very low driving rate and, therefore, a long refractory life. The methods of operating these latter vary considerably from plant to plant but there is in general some metallurgical work done, *e.g.*, the silicon content of the metal may be halved, and use is often made of the available heat to melt a useful proportion of scrap steel. In the inactive, or drum type, mixers on the other hand, virtually no metallurgical work is done, the object being merely to mix and to have a reserve of metal continually available. Some types it is true are fitted with gas burners but the fuel input is negligible compared with that on an open-hearth furnace. To be useful mixers must have a fairly high capacity, say a minimum of 250 tons, but they cover a wide size range, units of 1400 tons capacity not being unusual. Since their purpose is to hold metal at, say, 1300°C. until it can be used in the bessemer process it is vital that they be well insulated.

### (a) ACID LINED

This type of mixer, which is normally associated with an acid bessemer plant, acting as a holding vessel between the blast furnace and the converter, takes hematite iron of low sulphur and phosphorus content. Given correct thickness of insulation and expansion allowance, such mixers do not (soda ash excepted) present a particularly serious refractory problem. In some plants they are lined with silica bricks, which may be soft fired so as to reduce spalling, particularly in the door jamb regions, whilst in others, particularly in the United States, use is made of a dense fireclay brick, *e.g.*, the type recently developed for blast furnace hearths.

### (b) BASIC LINED

These mixers take basic iron from the blast furnace, frequently after soda ash treatment and hold it for subsequent use in a basic bessemer converter (fig. 204, p. 472). The roof of such mixers is often made of firebrick, say, of the 42 per cent. alumina type, the temperatures again only being of the order of 1300°C. The region in contact with metal and slag, however, must be lined with basic material and magnesite has



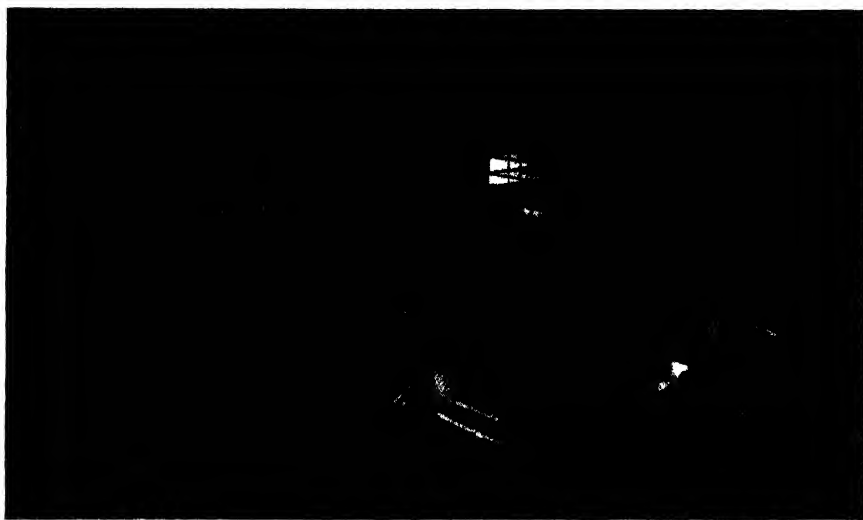
*Fig. 201. Acid bessemer converter (25-ton capacity) blowing.*



*Fig. 202. Acid bessemer converter (25-ton capacity) pouring into ladle.*



*Fig. 203. Basic bessemer shop (courtesy Stewarts & Lloyds Ltd.).*



*Fig. 204. Large inactive mixers serving basic bessemer plant (courtesy Stewarts & Lloyds Ltd.).*

tended to be the accepted refractory for this purpose. The use of this and similar materials immediately leads to problems not associated with the acid lined mixer, particularly if any appreciable amount of soda ash containing slag has been brought over from the ladle. Permeation of such slag into the bricks tends to promote shrinkage and, therefore, an unusual degree of volume stability is demanded. In addition basic refractories tend to have a very high conductivity and insulation, therefore, becomes a necessity. The problems involved in lining basic mixers have recently been reviewed by Parnham. His discussion refers particularly to a 600-ton type, in which 2 courses of magnesite are used with 1 course of backing firebrick, a course of compressible insulation and a final external course of insulating concrete (see fig. 205). From the known conductivity data Parnham calculates the

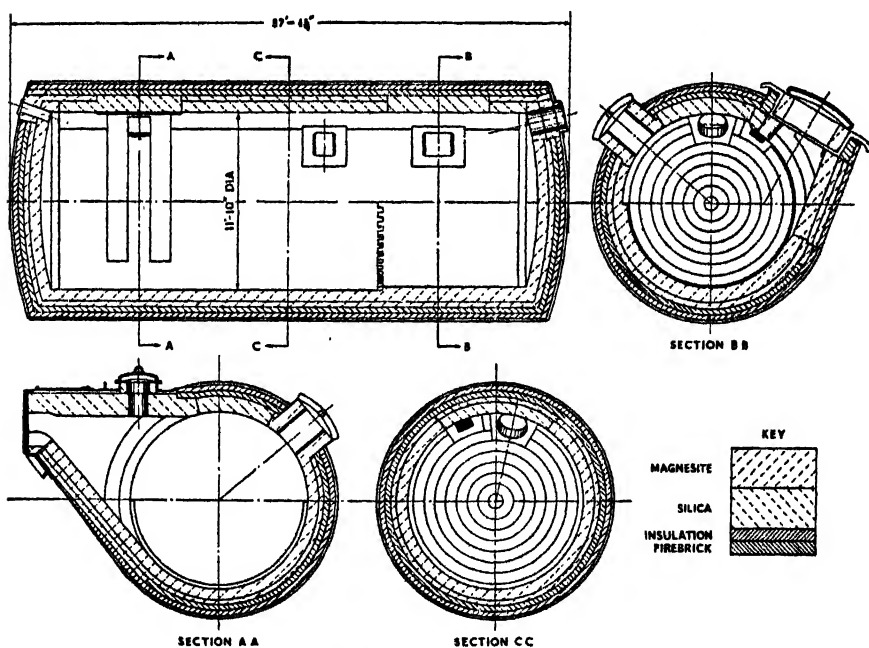


Fig. 205. Brick arrangement in 600-ton hot metal mixer serving basic bessemer plant (courtesy Richard Thomas & Baldwins Ltd.).

following temperature distribution, assuming a working face temperature of  $1300^{\circ}\text{C}.$ :

	°C.
Behind 11½ in. magnesite .. ..	1185
„ 4½ in. „ .. ..	1150
„ 8 in. firebrick .. ..	980
„ 6 in. compressible insulation ..	320
„ 2½ in. insulating concrete ..	146
Outside shell temperature .. ..	118

Even the low figure quoted above for the temperature of the shell is considered excessive in certain works, where it is stated that the actual temperature is so low that it can be comfortably touched with the bare hand. It is clear that such a temperature distribution, coupled with the high expansion coefficient of magnesite, will result in big differences in expansion between the inside and outside. Parnham, for example, suggests an expansion of 0.3 in. on the diameter of the steel case, but a corresponding expansion of 2.8 in. on the inner magnesite lining. Circumferential expansion may be handled by making use of compressible insulation all around the lining, or merely using it in the top half. Longitudinal expansion must also be catered for and this is usually done by putting in cardboard strips which burn out or steel spacers which are removed before the lining is heated up. Half per cent. or so of expansion can be handled in this way, the remainder being taken up by compressible insulation in the ends. In view of the difficulty of calculating movements in such a complex system and the great benefits, therefore, of first-hand experience, it is not surprising that sea-water magnesia linings installed in the early days of the Second World War were alleged to be inferior to imported material. Later comparisons, in which due attention was paid to correct expansion allowances, suggest that in actual fact there is little to choose between the two materials, provided they are both subject to similar conditions. Since the movements which occur during heating up are substantial, slow rise of temperature is considered essential; thus Parnham calculates that loads of 1000 lb. per sq. in. are necessary to give the observed 30 to 40 per cent. compression on the insulating material. The actual rates used vary between plants, but 15 days is considered a reasonable time for heating up a new installation, a rate 10 times as slow as that employed with an open-hearth furnace. The properties of the magnesite blocks employed (*see* Chapters III and IX) are essentially similar to those specified for use in open-hearth furnace bottoms, particular emphasis being given to low porosity, and minimum volume change on reheating at, say, 1500°C.

The condition of a mixer at the end of a campaign is shown in fig. 206, p. 477. Both the extent of the attack and the shrinkage of the inner lining due to slag absorption vary with the particular practice, *e.g.*,

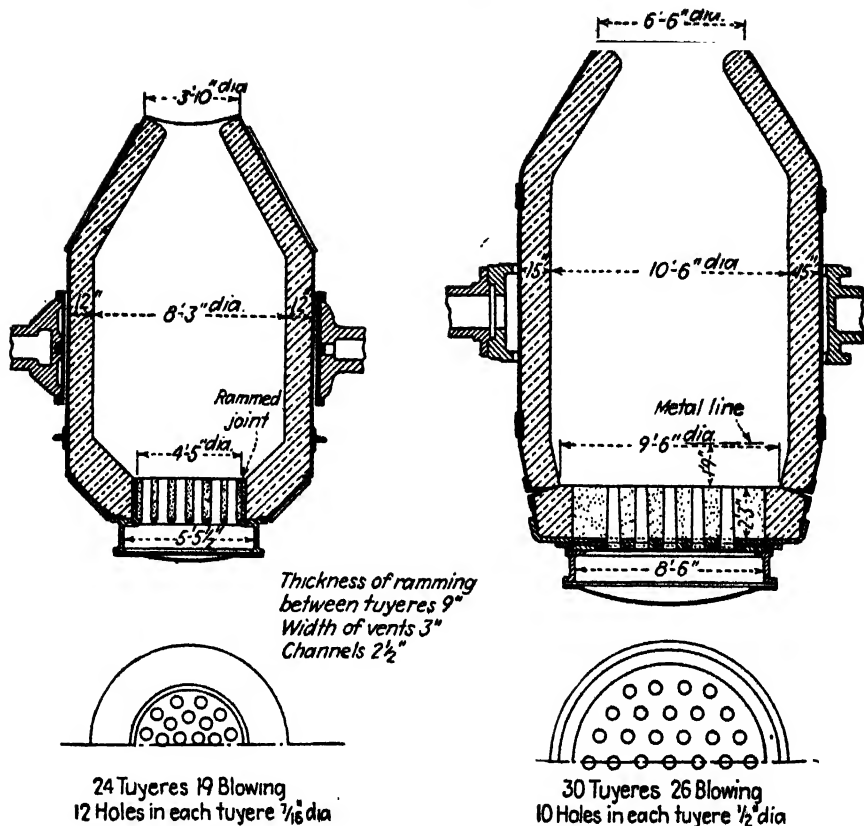


Fig. 207. Sections through 18 and 25-ton acid bessemer converters, showing general construction of bottom and vessel lining.

the precautions taken to remove soda ash. In some plants powdered lime is added (*see* Latour) but other mixer users are doubtful whether any advantage accrues from such treatment.

Several attempts have been made during recent years to obtain alternative type mixer linings, *e.g.*, Mayer and Knuppel state that the life of a mixer was increased by more than 50 per cent. by using chemically bonded magnesite bricks instead of the usual burned type, while Mayer, Gareis and co-authors tried dolomite bricks but found them to be less durable than magnesite.

## ACID BESSEMER CONVERTERS

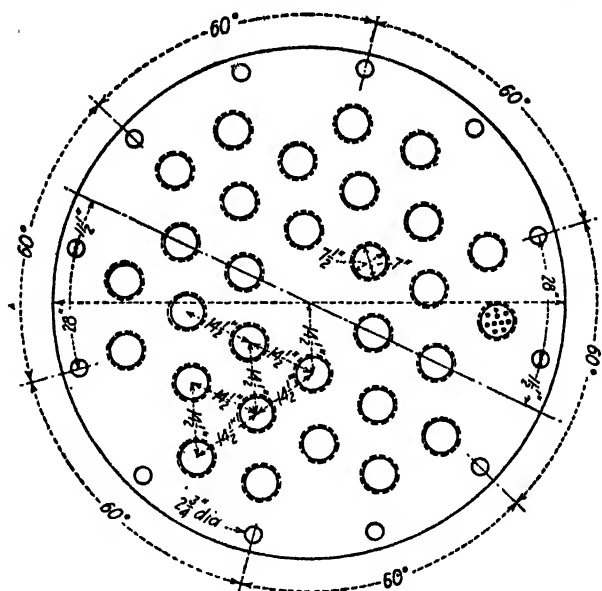
The acid bessemer shop usually contains at least 2 or 3 converters Fig. 201, p. 471, shows 2 characteristic 25-ton units, while fig. 207



shows sections through 18 and 25-ton converters, the smaller unit being fitted with a "cheese" type bottom and the 25-ton unit with a dish bottom.

#### (a) BOTTOMS

The amount of refractory material replaced with the "cheese" or cylindrical plug type unit is considerably less than with the dish type, but the practical advantages and in particular the ease of changing the bottom, have led to the almost exclusive use of the dish for large units. Fig. 208 shows a typical arrangement of tuyeres in a 25-ton



*Fig. 208. Distribution of tuyeres in 25-ton acid bessemer converter bottoms.*

acid converter dish bottom, from which it will be seen that the 30 tuyeres are distributed symmetrically with a view to obtaining even blowing over the whole bottom surface. The space between the tuyeres is usually rammed with a clay bonded firebrick aggregate, the amount of ramming used being reduced by employing fillers, *i.e.*, blocks of fired material, at intervals between the tuyeres. An alternative to ramming is the use of a slurry high in ganister. The actual number of tuyeres used and their distribution varies from plant to plant but for 25-ton converters it is usually between 25 to 35. For 10-ton converters about 20 tuyeres are employed.



*Fig. 206. Cobblestone type wear in magnesite lining of large inactive mixer (courtesy Stewarts & Lloyds Ltd.).*



(i) *Tuyeres*

Acid bessemer tuyeres are virtually always made from a fireclay grog mix, the amount of grog generally being about 10 to 20 per cent. and consisting either of crushed firebrick or firebrick with an admixture of ganister. Where ganister is added the object is principally the reduction of shrinkage and, therefore, of cracking in the kiln. From the point of view of refractoriness it would at first sight appear to be a retrograde step, but examination of used tuyeres shows the quartz is still there as quartz and that the tuyere is, therefore, behaving as a series of small fireclay and silica bricks side by side rather than a low melting point eutectic.

The length of the tuyeres is usually about 33 in. to 36 in. for a 25-ton converter. They are circular in cross-section but taper from 6 in. diameter at the working end to about  $7\frac{3}{4}$  in. at the bottom, where they bell-out to give a seal with the steel-bottom plate. With 10-ton converters the tuyeres may be only 2 ft. long while the number used is less than with the large converter. Plant histories show frequent changes in the number and size of tuyere holes but the range for 25-ton converters usually lies between 7 and 12 holes of  $\frac{7}{16}$  in. to  $\frac{1}{2}$  in. diameter. The use of a hole that only varies from a  $\frac{1}{2}$ -in. standard by very small amounts is presumably due to the fact that smaller holes would mean substantially bigger pressure losses and, incidentally, thinner and weaker piercing rods, while larger holes might lead to "boring", i.e., premature wear resulting from metal penetration into the mouth of the hole.

The manufacture of tuyeres is by no means an easy job, particularly with a material having such a high mould to fired shrinkage. It is not surprising, therefore, that almost all the tuyeres used in British and American plants are made by two manufacturers. In Great Britain tuyeres are normally made by dry mixing the graded batch in a pan mill, pugging with water, extruding a rough shaped tuyere, placing this in a lubricated brass mould and piercing it by means of steel rods inserted in what eventually becomes the bottom of the tuyere. The technique used must above all avoid any tendency of the holes to bunch together, since otherwise two or more may join in use and "boring" become excessive. After drying for several days on a steam-heated floor the tuyeres are trimmed and then fired to a temperature of approximately 1400°C.

The comparative properties of British and American tuyeres are summarised in Table XLVI, from which it will be seen that both are of the 38 per cent. alumina type and have melting points of the order of 1690°C. Their porosity is about 20 per cent. and their bulk density over 2.00 g.p.ml. Both types possess in addition a moderate permeability

TABLE XLVI  
PROPERTIES OF ACID BESSEMER TUYERES

	<i>British tuyere</i>	<i>American tuyere</i>
Chemical analysis (per cent.):		
SiO <sub>2</sub> .. .. .	57·8	53·7
Al <sub>2</sub> O <sub>3</sub> .. .. .	38·1	37·5
Fe <sub>2</sub> O <sub>3</sub> .. .. .	0·9	3·1
TiO <sub>2</sub> .. .. .	0·7	2·2
CaO .. .. .	0·4	0·5
MgO .. .. .	0·4	1·5
Alkalis .. .. .	1·0	Not done
Refractoriness, °C. .. .. .	1690	1690
Porosity, per cent. .. .. .	21·0	19·8
Bulk density, g.p.ml. . . . .	2·02	2·14
Apparent solid density, g.p.ml. . . . .	2·56	2·67
Permeability to air (parallel to but excluding tuyere holes), c.g.s. units .. .. .	0·07	0·10
Thermal shock resistance .. .. .	30+	30+

and a remarkably high thermal shock resistance. Although neither type can be considered as ideal, there is little doubt that the properties found and in particular the remarkable similarity between the two materials, arises from the survival of the fittest, the present product resulting from the gradual elimination of less successful types of material.

Attempts have been made to break away from the use of fireclay, *e.g.*, unfired magnesite and chrome-magnesite tuyeres have been tried both with and without a metal case and also plumbago (clay-graphite) tuyeres. So far the latter are the only ones reported to have shown markedly superior resistance to attack but their cost is far higher than that of fireclay tuyeres and is likely, therefore, to prevent their extended use.

#### (ii) *Fillers*

It is found helpful to place large blocks of refractory material at intervals between the tuyeres. The main function of these so-called "fillers" is apparently the mechanical reinforcing of the ramming mass, though there are other obvious advantages, *e.g.*, reduction in drying shrinkage and in the amount of moisture that must be removed from the bottom before it can be fitted to the converter. Where fireclay blocks are used they may be, say, 24 in. × 12 in. × 3 in. but excellent results

have also been obtained with triangular fillers of smaller cross-section which can be extruded and, not requiring any precise dimensions or smoothness, can be made relatively cheaply. The number and weight of fillers varies greatly from plant to plant, varying from nil to over 60 for the triangular type. Certain plants which do not use fillers as such do mix broken tuyeres with the batch rammed, a practice that is presumably more efficient in reducing drying shrinkage than providing reinforcing.

### (iii) *Ramming*

It has been suggested that acid bessemer bottoms could be built from a few large prefired blocks containing a uniform distribution of holes, as in the basic bessemer plugs. The cost of producing such blocks and the difficulty of obtaining the necessary thin joints between them, makes their use quite impracticable and all present acid bottoms are made by ramming around prefired tuyeres. Numerous materials could be used for this filling material, but experience has led to the almost exclusive employment either of clay-grog mixtures, or clay-ganister batches. Since the reduction of drying shrinkage and consequently of cracking is of paramount importance, most users prefer to employ batches high in grog (of the order of 50 per cent.) and a moisture content not more than is required to give consolidation of ramming. Certain plants do, however, make so-called "wet plugs", by pouring a mixture of two-thirds raw ganister and one-third fireclay into the gaps between the tuyeres. Such plugs crack on drying but the ease of making and the reasonable life obtained has led to their retention in certain works. An overall analysis of the ramming material used in a British plant shows a composition similar to that of the tuyeres ( $\text{Al}_2\text{O}_3$ , 37 per cent.,  $\text{SiO}_2$ , 60 per cent.,  $\text{Fe}_2\text{O}_3$ , 2 per cent.), whilst sieve analyses show gradings of the following type:

<i>Grading</i>	<i>Approximate</i>	
<i>B.S.I. sieves</i>	<i>Tyler sieve equivalents</i>	<i>per cent.</i>
$\frac{1}{2}$ – $\frac{1}{4}$ in.	$\frac{1}{2}$ – $\frac{1}{4}$ in.	10
$\frac{1}{4}$ in.–7 mesh	$\frac{1}{4}$ in.–8 mesh	10
7–25 „	8–28 „	30
25–72 „	28–65 „	20
72–150 „	65–150 „	10
Through 150 mesh	Through 150 mesh	20

It will be seen that the batch is very coarse, 20 per cent. being greater than would normally be employed in firebrick manufacture and 10 per cent. greater than  $\frac{1}{4}$  in. Although the procedure employed in preparing the batches is relatively crude the grading of the product shows a

remarkable degree of constancy. If, however, the best results are to be obtained it is important that both gradings and ramming density tests should be carried out at intervals. The moisture content of the material to be rammed pneumatically is normally about 8 per cent., the batch being fed in layers between the tuyeres and filler blocks inserted at intervals. In general the tuyeres tend to wear back first (*see* fig. 209, p. 487) and there is no particular point, therefore, in attempting to improve the ramming material. If, however, improved type tuyeres are developed more attention will have to be paid to the properties of the ramming.

Where "cheeses" are employed there is a similar arrangement of tuyeres and fillers but the ramming is carried out inside a cylindrical steel shell which is removed before the "cheese" is placed inside the drying oven. Ideal drying of such bottoms clearly calls for the use of a controlled temperature and humidity type drier, but the short life of plugs has led in most plants to drying arrangements that are far more severe and which ensure rapid drying, at least of the top half of the plug in a relatively short period. The plugs are actually pushed into a drying oven whose temperature is somewhere in the range 400° to 900°F. (204° to 480°C.). Under such conditions the water in the plug surface must evaporate extremely rapidly but is replaced in part by water migrating from the lower section of the plug. Even after 4 days of treatment at this relatively high temperature the lower layers are still moist, as can be readily demonstrated by pushing a cold steel rod down into a tuyere or vent hole and observing the condensation. To dry such a plug with the care that would be given to it were it a large shape made in a brickworks would demand several weeks and consequently call for an extravagantly large drying plant. That the present compromise is effective is presumably due in the main to the high proportion of coarse pre-fired material in the batch, which tends to keep the latter open and also results in a relatively small drying shrinkage.

(iv) *Vessel joint*

Before bolting the dried bottom to the converter a layer of cement—usually a ganister-clay mixture—is placed around the circumference. On tightening up this spreads out to give a reasonably tight seal.

(v) *Bottom life and costs*

An American firm recently claimed a record life for a bottom of over 100 heats, achieved in part by dummieing-up, *i.e.*, blocking off, an abnormal number of failed tuyeres. With a blowing pressure of, say,

25 lb. per sq. in., a blowing area of 60 sq. in., a bath depth of 20 in. and a blowing volume of about 30,000 cu. ft. per minute, a 25-ton charge can be blown in approximately 10 minutes. Given these conditions and a favourable manganese/silicon ratio in the metal an average plug life of about 40 blows can be obtained. If, however, there is a serious lack of blowing power, as was the case in one plant where a 25-ton converter was blown with the same engine as had previously been used for a much smaller unit, then plug life is bound to suffer and 16 to 20 heats become a more typical figure. Under such conditions the refractories cost may well be doubled.

The appearance of a plug at the end of a run is illustrated in fig. 209. It will be seen from this that the wear is by no means even, being, with this particular plug, far more severe in the areas adjacent to the tuyere ends. Periodically one tuyere wears back at a rate out of all proportion to that shown by the other tuyeres. The explanation of this so-called "boring" is by no means clear, but is generally associated with two tuyere holes being closer together than normal and in consequence running together to form a wide throat (*see* fig. 210, p. 488). Once such a cavity has formed metal will fall into it due to the localised reduction in effective blast pressure and will then be set in rapid motion by the air blast, in much the same way as a ping-pong ball spins on a jet of water. Rapid oxidation of this whirling mass of metal will lead not only to very high local temperature but also to the production of large amounts of corrosive oxides and, therefore, also to a rapid rate of solution of the tuyere. The rate at which such boring can take place is nothing less than phenomenal, a tuyere sometimes being penetrated at the rate of 1 in. per minute. It is suggested periodically that boring is associated with the presence of inferior refractory, but this is not supported by comparisons made of the longest and shortest tuyeres in a number of worn out bottoms. It will be seen from one such comparison made in Table XLVII that there is in fact little to choose between the physical properties of long and short tuyeres. Fortunately the failure of one such tuyere does not put the bottom out of action, since the offending tuyere can be dummied off with clay, or removed entirely and a new one inserted.

Analyses of slag removed from tuyere ends suggest that most of the chemical attack is due to oxides of iron and manganese. It would, therefore, be logical to consider such attack in terms of the equilibrium diagram for the quaternary system  $\text{FeO-MnO-Al}_2\text{O}_3\text{-SiO}_2$ . This has unfortunately not yet been worked out, but a study of the corresponding diagrams for alumina and silica in combination with FeO or MnO separately, shows that an addition of only 20 to 30 per cent. of these



TABLE XLVII

A COMPARISON OF THE PROPERTIES OF THE LONGEST AND SHORTEST TUYERES IN A USED ACID BESSEMER PLUG

	<i>Longest tuyere from plug</i>	<i>Shortest tuyere from plug</i>
Porosity, per cent. . . . .	18.0	18.1
Bulk density, g.p.ml. . . . .	2.08	2.06
Apparent solid density g.p.ml. . . . .	2.53	2.52
Permeability to air (parallel to but excluding the tuyere holes), c.g.s. units . . . . .	0.093	0.056
Permanent linear change on reheating— 1 hour at 1400°C. . . . .	No change	0.6% (sh.)
Refractoriness-under-load—50 lb. per sq. in.— rising temperature:		
Initial softening . . . . .	1350°C.	1140°C.
Rapid softening . . . . .	1430°C.	1400°C.
Fail (10 per cent. deformation) . . . . .	1640°C.	1580°C.
Thermal shock resistance . . . . .	30+	30+
Melting point of slag on tuyere end . . . . .	1380°C.	1360°C.

oxides is sufficient to lower the melting point of a 38 per cent. alumina firebrick to about 1100°C. Such a reaction product could be expected to be extremely fluid at bessemer operating temperatures. The action at the tuyere ends will tend to be greater than between the tuyeres, because it is at these positions that the oxides are formed and turbulence is also a maximum. Sections cut through acid bessemer slag samples show the presence of "stones", *i.e.*, quite large undissolved particles of refractory, which must have come from the bottom. Furthermore a determination of the alumina content of the slag (usually about 4 per cent.) offers a fair indication of the amount of material dissolved during any particular blow.

Other factors that may be expected to affect bottom life, are the amount of scrap charged, together with the method of introducing it, and the periodicity of use. Where, as in some plants, heavy rail sections slide down a chute and dive into the molten metal at considerable speed, some mechanical damage is bound to result even though attempts are made to minimise the impact by using trap doors at the end of the scrap chute. No definite information appears to be available on the effect of standing time on converter life. It can, therefore, only be stated that on the one hand frequent heating and cooling is liable to

result in spalling or general loosening of the plug material, but on the other hand the steeper temperature gradient in an infrequently used bottom may tend to reduce the rate of wear.

#### (b) VESSEL LINING

Most American converters are lined with some form of sandstone. In some plants quite good results have been obtained with the commoner grades, known for example as "ironstone" and "firestone", but the best results appear to be given by special micaceous schists, tests on one sample of which are given in Table V (Chapter II). This material not only shows a high durability but can by reason of its laminated structure be readily cut into rectangular-shaped blocks. On the other hand British converters are usually lined with relatively soft-fired silica bricks. It is very surprising at first sight that either of these materials is satisfactory, since the conversion of the large amounts of raw quartz present to cristobalite might be expected to result in substantial expansion followed by slabbing away of the working face. Such expansion doubtless does occur but in a cylindrical vessel apparently leads in the main to a tightening of the structure rather than to fracture, whilst the initial heating-up is made far safer by the absence of a large proportion of cristobalite, such as would be present in a hard-fired silica brick. In some plants new linings are initially protected with a layer of 2 to 3 in. of ganister, but in others unprotected bricks are standard practice. The life of linings in acid converters varies greatly from plant to plant and is clearly dependent on the amount of patching done; a life of 400 blows, or roughly 10 times the bottom life has, however, been quoted.

### BASIC BESSEMER CONVERTERS

The general layout of a basic bessemer shop (*see* fig. 203, p. 472) is similar to that for the acid converter, there being usually a series of converters in line with one or more large cylindrical mixers.

#### (a) BOTTOMS

Bottoms used in basic converters would be described by acid bessemer men as "cheeses", *i.e.*, consisting of a cylindrical mass of refractory material, the air holes either being scattered over the bottom in a regular manner or use made of tuyeres as in the acid process. For a 25-ton converter the bottom has a diameter of about 7 ft. and a depth of about 3 ft. The 45-ton size converter, such as is employed in certain German plants, naturally calls for a proportionally larger bottom. Broel gives a diameter for a 45-ton unit of 10 ft. but a bottom thickness

similar to that for a 25-ton unit, *viz.*, 3 ft. 3 in. This particular bottom contained 348 holes, each of a diameter 14.5 to 15.0 mm. (0.57 to 0.59 in.) and a total blowing area of 17.4 sq. cm. (2.7 sq. in.) per ton of metal charged. It is interesting to note how close the hole size used in this converter and in the 25-ton British converter (271 holes of 13 mm.) is to that employed in the acid process.

### (i) *Raw materials*

The essential constituents for rammed bottoms are doloma and tar. It has been stated in the past that the doloma should be softer fired than for open-hearth work but it may well be that this preference merely arises from the ease with which a satisfactory grading is obtained when a softer material is employed. The essential feature of the tar is that it should be water-free, though other properties are sometimes specified. A fair impression of the type of tar considered desirable may be gained from the following tests carried out on a sample from a British plant:

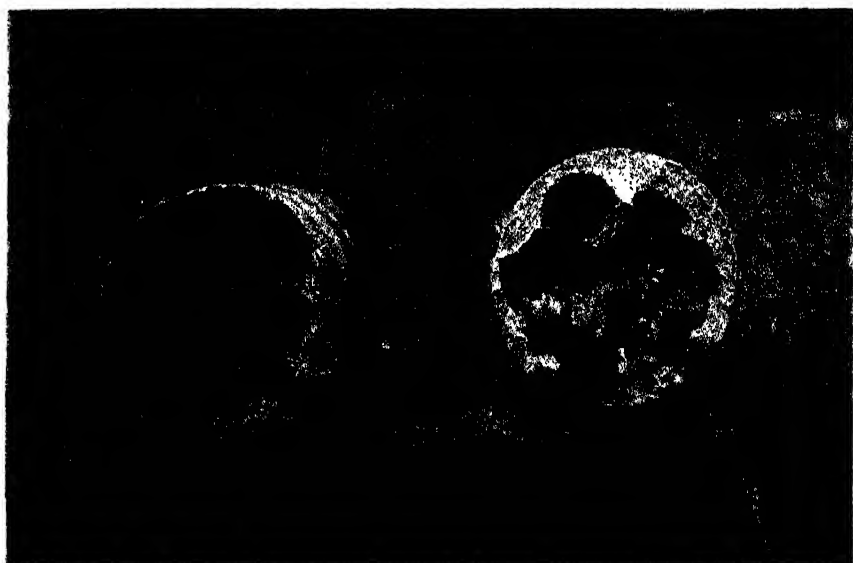
Specific gravity	1.197	Tar acids	2.00% vol./wt.
Viscosity	20 sec.	Free carbon	10.48% wt./wt.
Naphthalene	0.68% wt./wt.	Pitch	60.67% wt./wt.
Distillation range:			
Water	Traces	Upto 300°C.	9.3% by wt.
Up to 270°C.	8% by wt.	300°-350°C.	22.0% by wt.
Conradson carbon residue test:			
Tar as received	27.86%	Pitch as made	46.00%

This material was supplied against a specification giving a maximum free carbon of 10.5 per cent. pitch content of 60 to 63 per cent., water—trace only, and naphthalene less than 1.5 per cent. The amount of tar used varies with the technique employed, *e.g.*, the fineness of grind and method of ramming, but is usually between 7 and 12 per cent.

The grinding of the material to form a mass having a high packing density follows the general principle employed for basic open-hearth furnace material (*see* Chapter IX). The corresponding procedure for the bessemer process was fully described by W. C. Bell in the Iron and Steel Institute Special Report No. 33, while further information was given by W. L. Kerlie in the Iron and Steel Institute Special Report No. 35. Thus in one plant the doloma from a cone crusher is fed to a pan mill, the desired amount of tar added and milling continued for 15 to 20 minutes for converter lining material but 30 minutes for the doloma required for bottoms. To facilitate mixing the tar is added at a temperature of 90° to 100°C. This, together with the heat released by



*Fig. 209. Worn-out bottom of 25-ton acid bessemer converter. Note the localised attack at the tuyeres.*



*Fig. 210. Used tuyeres from acid converter bottom. Note the irregular wear and signs of "boring".*

grinding and the expected heat losses, results in a final batch temperature of 60° to 65°C. Typical gradings of the material as fed to the mill and after milling with tar are given below, together with the corresponding packing densities and tar contents:

<i>Grading B.S.I. sieves</i>	<i>Crushed doloma (dry)</i>	<i>Milled doloma (tarred)</i>
+ $\frac{1}{4}$ in.    ..    ..    ..	Nil	Nil
$\frac{1}{4}$ – $\frac{1}{2}$ in.	5.0 per cent.	6.4 per cent.
$\frac{1}{2}$ in.–7 mesh    ..    ..	33.7    "	32.6    "
7–25    "    ..    ..	24.4    "	22.0    "
25–72    "    ..    ..	14.9    "	13.7    "
72–150    "    ..    ..	8.8    "	7.9    "
150–300    "    ..    ..	13.2    "	6.6    "
–300    "    ..    ..	—	10.8    "
Packing density. g. per ml.    ..	2.14	2.43 @ 60°C.
Loss on ignition, per cent.    ..	2.30	8.20
Tar content, per cent.    ..	—	5.9

### (ii) *Tuyeres*

In some plants fired magnesite tuyeres or preformed tarred doloma tuyeres are employed. Welter for example claims that a 5 per cent. improvement can result from the use of doloma tuyeres instead of the normal perforated bottom. In the practice he describes, 40 to 50 tuyeres are employed, each 18 cm. (7.1 in.) in diameter and 75 to 95 cm. (29.5 to 37.4 in.) high. Each tuyere contains 4 holes and is placed in position in the bottom plate without any preforming. It is said that such tuyeres can be pressed at the rate of 4 minutes per tuyere and only 2 to 3 hours are, therefore, required to make sufficient for one bottom. The tarred doloma used between the tuyeres has a low tar content when pneumatic ramming is employed but may have a high tar content and be merely poured into position. The bulk density of tuyeres made in this way is stated to be similar to that of bessemer side wall blocks of the same composition and it is claimed that the rate of erosion at the tuyere hole is less than with a monolithic bottom. The record at this plant for a normal bottom was given as 88 blows compared with 96 for a tuyered unit. Where preformed tuyeres are employed they represent 55 per cent. of the total bottom weight, a much higher proportion than in the acid bessemer process.

### (iii) *Production of bottoms*

There have been many new developments in bottom technique during recent years, but some form of machine is normally employed

to do the compacting. Fig. 211 shows a typical installation designed to stamp the bottom in a series of layers, the movement of the stamping head being automatically controlled to give each part of the bottom an equal amount of ramming. As the bottom is rammed, steel needles are automatically thrust up through it from below and penetrates the surface as the process reaches completion. The thickness of the

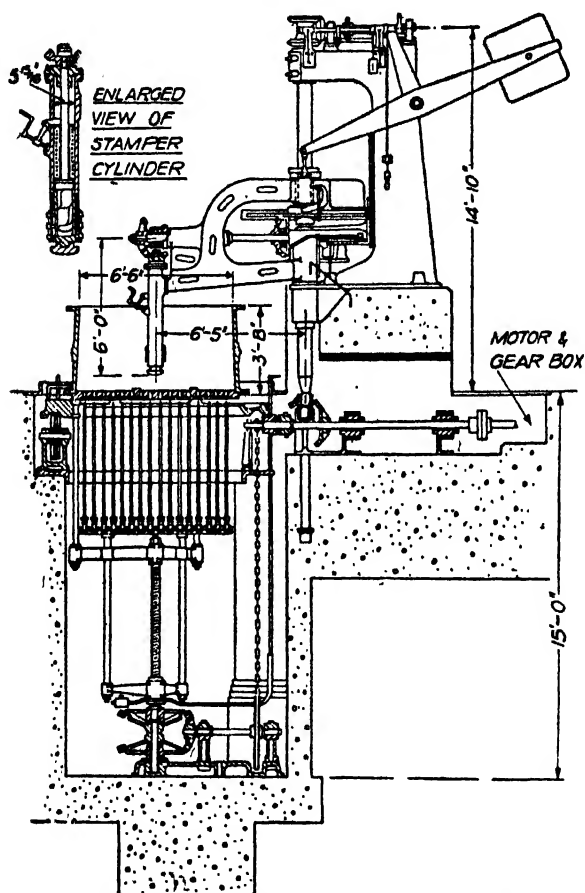


Fig. 211. Machine used for making basic bessemer converter bottoms.

layers rammed is partly governed by the capacity of the pan mill, also by the degree of compacting that can be obtained through a given depth of material. Care is always taken to roughen each rammed surface in order to ensure union with the layer above. If this is not done then slabbing away of the bottom may occur due to weakness at the laminations.

When the needles have been withdrawn, wooden dowels are driven into the tuyere holes to prevent them from becoming blocked during the subsequent firing period. The bottom, still in its metal case, is covered with a heavy circular casting, to prevent swelling of the monolith and is then placed in a so-called drying oven. The term "drying" is a misnomer, in that there is no appreciable loss of water, while the final temperature ( $500^{\circ}$  to  $750^{\circ}\text{C}.$ ) suggests the process could better be described as light firing. The heating normally takes place in some type of tunnel kiln, the bottoms entering on bogies and taking a period of the order of a week to complete the firing schedule. The optimum rate of heating is that at which the tar is carbonised without undue loss due to burning or drainage into the lower portion. Incidentally the firing of tarred doloma in a reducing atmosphere has the effect of increasing the durability in storage, such bottoms if left in their case being quite usable after a period of months, where ordinary doloma would have long since failed by hydration.

More recently considerable use has been made of vibrators, *e.g.*, of the eccentric type, to do the compacting. The equipment required has been described in some detail by Latour and Schoop, who studied the effect of the power input to the vibrator and the tar content and various other factors, on the durability of the plugs in service. Here again compacting is, of course, carried out in a series of layers. At an early date extensive studies were also made by Eilender and Schoop on the effect of grading, tar type and ramming, on porosity of the final plug.

Among the other devices employed for increasing bottom durability are the insertion of metal tubes in tuyere holes, and the use of both vertical and horizontal reinforcing. Incidentally Welter claims that inclined tuyere holes led to reduced "spitting", but does not claim any increase in plug life.

#### (iv) *Vessel joint*

As might be expected tarred doloma is also used to form the joint between the doloma bottom and the doloma side walls. In order that this cement be able to flow freely and, therefore, form a tight seal, the doloma is ground with an unusually large percentage of tar (16 to 20 per cent.).

#### (v) *Bottom life*

A typical life for a 25-ton basic bessemer converter bottom is 40 to 50 blows. Broel gives a figure of 64 charges for a 40 to 45-ton converter, compared with a side wall life in the same vessel of 380. These latter results were obtained with an initial metal temperature of  $1180^{\circ}\text{C}.$ , a



pouring temperature of  $1450^{\circ}$  to  $1470^{\circ}\text{C}$ . (uncorrected) and a maximum blowing pressure of 33.8 lb. per sq. in. The blowing time for a 35-ton charge was about 18 minutes and the time for one charge to the next approximately 30 minutes. The total consumption of refractories in the 25-ton basic British unit is given as 50 lb. per ton of steel, of which 35 lb. was used as tarred doloma and 15 lb. as semi-stabilised dolomite bricks.

The longer life of the vessel lining (*see* later) generally results in 4 or 5 bottoms being fitted for every lining. When the first bottom wears thin, the bottom plate is removed and after a few holes have been drilled near the joint, the old bottom is pushed in. The joint is then cleaned up and a new bottom placed in position, the space between it and the vessel being filled with tar-doloma slurry. To prevent any slurry blocking the tuyere holes the latter are filled with soft clay, which can be rodded out prior to putting the converter into service. The slag formed in this process is entirely different from that with acid converters, Broel, for example, giving the following range of analyses:

	Per cent.
Total iron .. .. .	8-11
Manganese .. .. .	4.5
Lime .. .. .	48-52
Magnesia .. .. .	1.5-2.5
Phosphorus pentoxide .. .. .	19
Residue .. .. .	7-9

Most of the lime comes from the lime charged with the metal, no such addition being made in the acid process. The magnesia comes from the

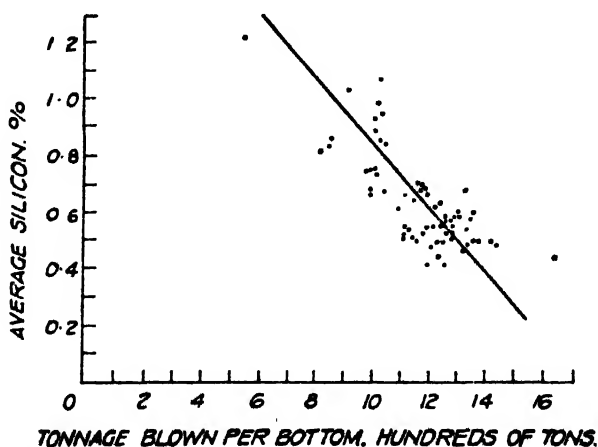


Fig. 212. Effect of silicon on basic bessemer bottom life (after W. C. Bell).

bottom and side walls, and can be used as an index of the amount of corrosion occurring during any particular blow.

One factor which it is generally agreed has a marked effect on bottom life, is the silicon content of the iron. Thus Bell gives the relationship shown in fig. 212, for bessemer bottoms at the Corby plant of Messrs. Stewarts and Lloyds. Reference has also been made by a number of authors, in particular Metz, Gregoire and Decker, to the protective action of residual carbon in the doloma mass. They suggest that the presence of such carbon reduces iron oxide that would otherwise attack the doloma, and that the failure of the latter occurs by gradual decarburising of the surface layers followed by penetration and solution. Metz also suggests the desirability of having present a high proportion of coarse-grained doloma.

#### (b) VESSEL LINING

##### *Monolithic type*

Until recently basic converter linings were either rammed with a tarred doloma mass (*see* fig. 213), or partially built of similar material pressed in brick form and used unfired. As will be seen later this practice is giving place in certain plants to the use of fired bricks. The actual shape of the converter is similar to that in the acid plant being normally circular in cross-section but also built with an oval design. Both the material used for ramming linings and for preformed blocks is similar to that employed in bottoms, though such things as the tar content may be slightly adjusted in order to facilitate production. The blocks are formed in large hydraulic presses with a loading of about 5000 lb. per sq. in. They are usually installed soon after moulding, but may, if desired, be prefired in a manner similar to that employed with the bottoms. Before a new lining goes into steel production it is "broken in" by charging hot coke and subsequently lime and about a ton of molten iron and blowing until these are completely converted into slag. A selected composition of such a slag is as follows:

FeO	..	..	30 per cent.
Fe <sub>2</sub> O <sub>3</sub>	..	..	20 "
CaO	..	..	25 "
MgO	..	..	15 "
SiO <sub>2</sub>	..	..	6 "
Al <sub>2</sub> O <sub>3</sub>	..	..	4 "

As the result of this treatment the lining of the converter becomes coated with slag and is in a condition suitable to receive the first full charge of metal.

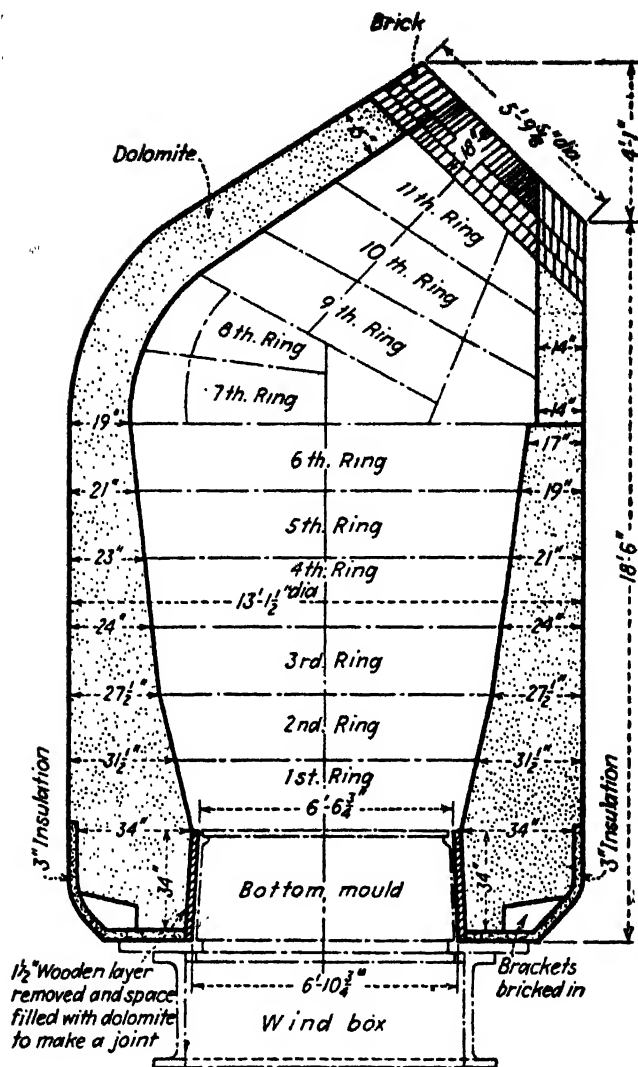


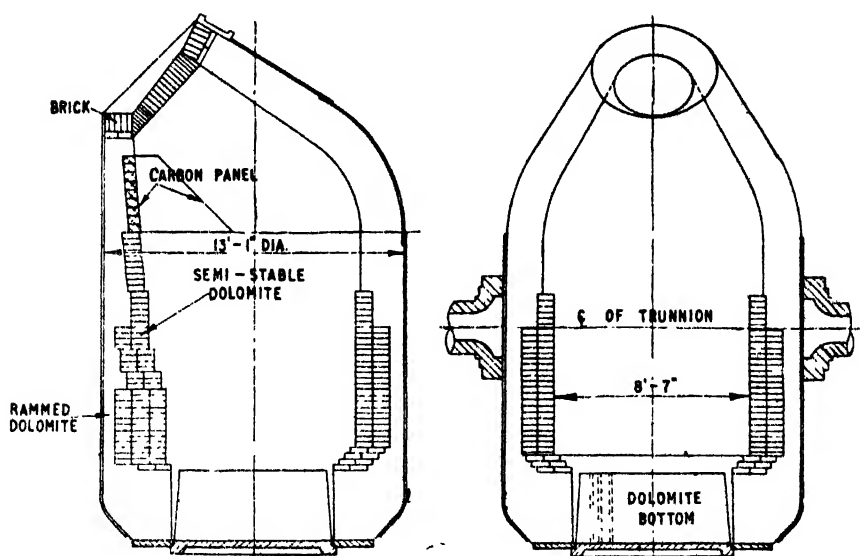
Fig. 213. Section through 25-ton basic bessemer converter, showing monolithic dolomite lining.

Comparatively little has been published on the factors determining converter lining life, but it would appear probable that both chemical attack and abrasion play a part. Thus silicon content which has been shown to be critical with bottoms, has also a marked influence on lining life. The wear tends to be greatest in a region about one-third up

the converter from the bottom to the mouth, where the lining tends to "bell-out". There can also be serious wear at the nose. A typical life for a monolithic lining in a 25-ton converter is 160 to 250 blows, though considerably higher lives are obtained with low silicon irons.

### *Brick type*

The very successful results obtained by Messrs. Stewarts and Lloyds at Corby with vessel linings built from semi-stable dolomite bricks of the type described in Chapter IV (*see* Table XIV) have been described by Bird. In spite of increasing silicon content in the metal, lining life at this plant has been increased from a minimum of 4100 tons (170 blows) in 1942, to over 6900 tons (266 blows) in 1952. This improvement is attributed in the main to the changeover to semi-stable bricks. Several arrangements were tried out before the present design shown in fig. 214, was developed. It will be seen that in this latest arrangement the



*Fig. 214. Sectional views of eccentric lining employing semi-stable dolomite bricks (after D. Bird).*

refractory at the base of the back wall, which is the position of greatest wear, is 33 in. thick, of which 18 in. consists of brick and the remainder of rammed doloma. The brick thickness is maintained round the vessel but the rammed doloma is reduced to 9 in. at the sides and 7 in. at the opposite wall. Incidentally one result of this design is that the bath is

shallower than normal. When a new lining is to be installed the old one is removed right back to the shell, except for the bottom zone where the rammed doloma is thickest. The dolomite bricks are then laid dry without a former, the contour being determined by measurement from the shell. No expansion joints are left between the bricks, which act as a shuttering for the tar-doloma mixture rammed between them and the case. Although the time for relining is a few hours longer than with the monolithic design and the bricks considerably more expensive than rammed doloma, the increased production resulting from longer and more regular lining life justifies the change, particularly since the shallower bath makes for improved steel quality.

### SIDE BLOWN CONVERTERS

The use of side-blown converters became so general in Great Britain during the Second World War that a special sub-committee was set up in 1943 to tackle the problems associated with them, and in particular those related to refractories. The committee started work by issuing a questionnaire to 22 firms and analysing the results in 3 main groups according to capacities: *viz.*, 23 cwt., to 30 cwt., 40 cwt. to 47 cwt., and 50 cwt. to 60 cwt. The answers, which are summarised in the Journal of the Iron and Steel Institute (*see* bibliography), showed that for smaller vessels there was a general preference for monolithic linings, but that the larger vessels frequently used brick. In spite of the wide differences in size and practice, the number of heats blown during a run were substantially constant. The actual cost of refractories is, however, appreciably less per ton of steel for the larger converters. The location of the wear is clearly shown by the numerous illustrations given in the report. It is demonstrated that it is most extensive on the blowing side, particularly in the region of the tuyeres and is more pronounced on the upper than on the lower side. Wear also takes place on the teeming side, mainly from the junction of the body with the nose and thence to the nose end. Many operators have attempted to obtain more efficient use of refractories by thickening them in the critical areas. Certain plants were able to show a substantial improvement in lining life by charging sand into the converter with the iron, thus providing a flux for the iron-oxide produced in the early stages of the blow. Such additions must be added to the refractories' cost but may well be economical in view of the low price of sand, particularly since the material used is often old core sand which would otherwise be thrown away.

The comparison of lives between plants is complicated by the extent of hot patching, which varies greatly from one plant to another. In

some works the patching material is mixed to a cream and applied immediately after tapping, whilst at others the converter is allowed to cool to 600°C. and the patching material rammed in position.

The monolithic materials used consist essentially of quartz in the form of ganister, quartzite or sand, bonded with varying proportions of clay. Where bricks are employed they are usually of a quality similar to that used in open-hearth furnace roofs, though special silica bricks with high iron oxide content (about 3 per cent.) are also employed.

Chemical analysis of a number of monolithic linings shows a silica content from 88 to 94 per cent. and an alumina content of 2 to 6.5 per cent. The remainder of the analysis is similar to that of silica brick except that the lime tends to be on the low side—1 per cent. or less. The physical properties of such materials vary widely with the raw material used in their manufacture, some showing a maximum grain size in the 1 to  $\frac{1}{2}$  in. range and others consisting largely of extremely fine material—less than 150 mesh. The refractoriness tests showed values consistently in the region of 1680°C. which is 30°C. below that of a good silica brick but characteristic of the refractoriness of the working face of a used brick.

Drying and firing tests showed most of the materials to have a negligible drying shrinkage, but a firing expansion varying from 1 to 3 per cent. after 2 hours at 1410°C. Harder firing (at 1530°C.) caused some of the materials to shrink, whilst others continued to expand. The porosity of the fired testpieces, which may, however, be higher than would be expected with the actual lining, are generally of the order of 25 to 30 per cent.

Studies were also made of the changes occurring in service, both with brick and monolithic material. Where silica blocks are used a zone structure was obtained similar to that seen in the open-hearth furnace but on a much smaller scale, the whole sequence of zones being confined to the hotter 2 in. of brickwork, presumably due to the steeper temperature gradient in the converter lining, which is in turn connected with its intermittent use. There was, as might be expected, a substantial pickup of iron oxide in the working face and a corresponding drop (to about half) in the porosity.

Among the recommendations made by this Committee were more rapid drying of converters by means of gas flares but the avoidance of heating up by blowing of coke, since this practice was found to lead to excessive rates of temperature rise and might, therefore, well be responsible for spalling.

Cress reached the conclusion that neither silica brick nor refractory sandstones were particularly desirable, better results being obtained

with clay bonded ganister. In order to achieve both high density and high refractoriness this author recommends the use of 50 per cent. of  $\frac{1}{4}$  in. ganister, 30 per cent. of  $\frac{1}{4}$  in. ganister, 14 per cent. of silica flour and 6 per cent. of bentonite. The use of bentonite instead of fireclay is considered worth while, because a substantially smaller quantity is needed to get strength and there is, therefore, less drop in melting point due to the alumina addition.

## OTHER PNEUMATIC PROCESSES

Comparatively little has yet been published regarding the refractories employed in the new processes, such as the L-D, the oxygen rotor and the Haspe oxygen/steam. In the first of these no tuyeres are involved other than the water-cooled copper lance and, as might be expected, the doloma consumption is, therefore, relatively low. Since the bottom life in the L-D process is excellent there is no need to make the bottom removable. The actual procedure for lining doubtless varies from one plant to another, Schaden, for example, suggesting a permanent lining of magnesite bricks and an inner lining of pressed tarred-doloma bricks.

The refractory problems associated with oxygen-steam blowing have been described by Kosmider in his 1950 paper. In the early trials an attempt was made to improve on the normal doloma plug by using steel tube inserts, but even with 50 per cent. of oxygen in the blast excessive wear occurred after only a few blows, due it is thought to localised attack by ferrous oxide on the doloma. Later tests in which the steel pipes were replaced by copper pipes gave far better results even with oxygen concentrations as high as 92 per cent. Indeed with this new technique a life of 50 to 80 blows was obtained with this particular type of converter and process. Just why the use of copper tubes should make such a difference is not clear, though their high conductivity doubtless helps by reducing the length of pipe melted off at the working face and, therefore, the depth of unsupported doloma.

The lining of the oxygen blown rotors presents a rather more serious problem, since wear is heavy – comparable with that in an open-hearth furnace making low carbon steel – and fettling is, to say the least, difficult. Fairly frequent replacement of the lining is likely to be necessary, which would in turn demand additional vessels. The present refractory is tarred doloma, though the inconvenience of frequent shut-downs might justify the use of special linings, say of magnesite.







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## Chapter XII

# THE ELECTRIC STEEL PLANT

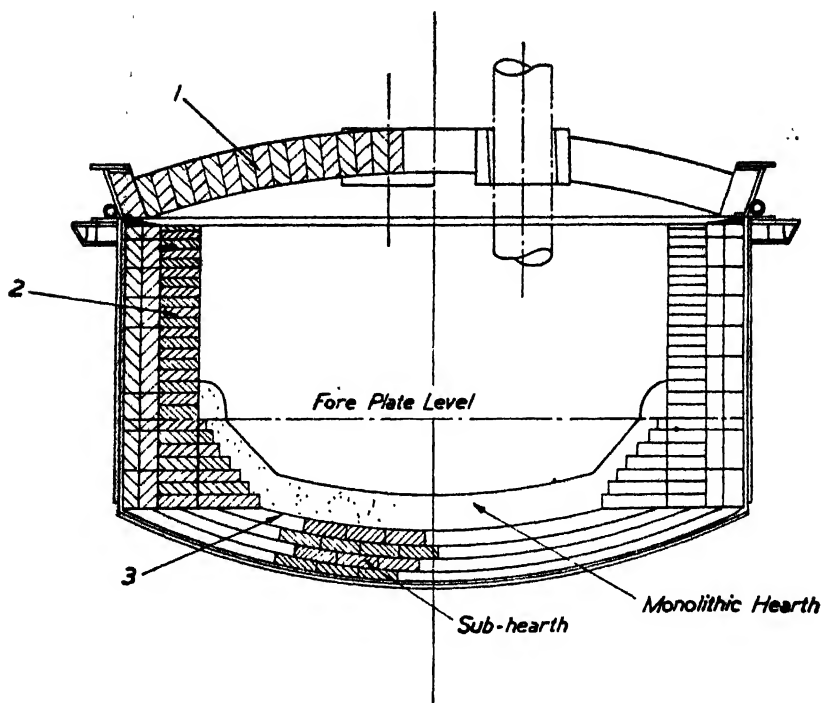
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THE ARRIVAL OF ATOMIC ENERGY is likely to increase the proportion of steel made in electric furnaces. In certain parts of the world, where scrap is more readily available than pig iron, the electric furnace is already competitive with the open-hearth or bessemer, but for the most part it is appreciably dearer to operate and is, therefore, only used for the production of special, *e.g.*, stainless, steels, or as reserve capacity that can be brought into operation at relatively short notice. Although it has been shown, *e.g.*, by Westinghouse, that where avoidance of contamination is vital, metals can be induction heated in space by making use of the electromotive forces between twin coils, all production furnaces still require refractories and, moreover, refractories capable of withstanding rapid heating and cooling, high temperatures and severe slag attack. The two main types of electric furnace employed are:

- (1) The Arc Furnace, almost always of the "direct" type, in which 2 or more electrodes (usually 3) are caused to arc to the scrap or molten bath. In the "indirect" process, the current passes from one electrode to another and the steel is heated by radiation.
- (2) The Induction Furnace, which consists essentially of a fired or unfired crucible surrounded by a water-cooled coil through which passes an alternating current, the metal being heated up by the eddy currents produced in it. If the coupling is efficient—and this generally means a thin lining—and the insulation of the crucible adequate, then melting occurs.

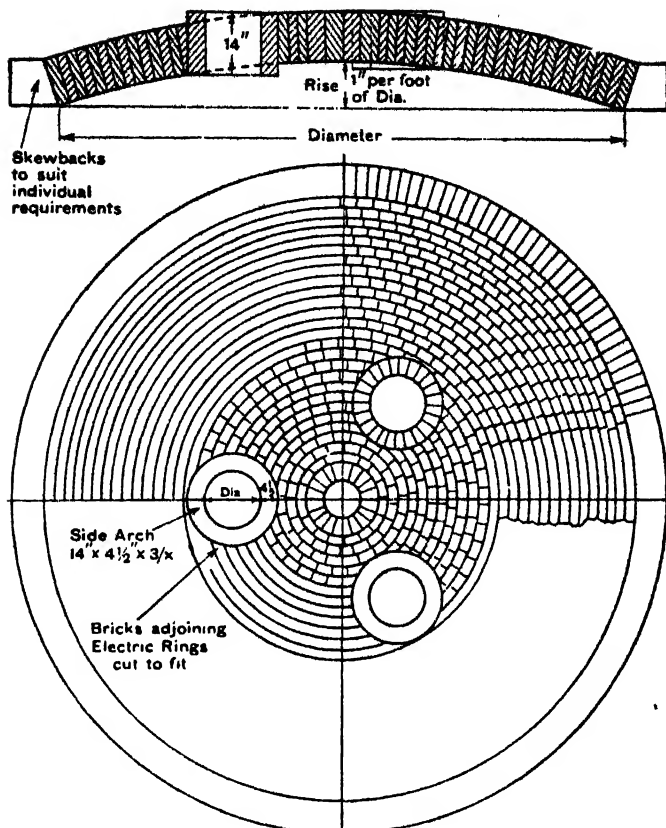
## THE BASIC ARC FURNACE

Such furnaces vary from  $\frac{1}{2}$ -ton or less in size to units of 200-ton capacity. The smallest units are mainly employed in foundries or for experimental work, whilst furnaces of 100-ton capacity and upwards are still comparatively rare, the great proportion of furnaces lying in the 10 to 100-ton capacity range (*see* figs. 215, p. 509, and 216, p. 510).



*Fig. 218. Arrangement of refractories in basic arc furnace: (1) Silica roof; (2) Metal-case magnesite bricks or tubes, chrome-magnesite, or semi-stable dolomite brick side walls; (3) Dolomite or magnesite monolithic hearth with stabilised dolomite or magnesite brick sub-hearth.*

Most modern arc furnaces are designed for 3-phase, 60-cycle operation. The voltage used varies with the size, but may, for example, be 90 to 220 volts on a 10-ton furnace and 90 to 325 volts on a 60-ton furnace. The current passed is extremely high, being about 10,000 amperes per electrode on a 10-ton unit and nearer 30,000 on a 60-ton furnace. The electrodes are themselves refractories, in that they are extremely difficult to melt, consisting in general either of graphite or amorphous carbon. Since the conductivity of amorphous carbon is only a quarter of that of graphite electrodes, the former type of electrode must be substantially larger to carry the same current without undue heating. Thus for 10-ton furnaces the sizes are about 12 in. and 20 in. respectively, whilst on a 60-ton furnace they would be nearer 20 in. and 35 in. in diameter. In most furnaces there are 3 electrodes, which pass vertically through the central region of the roof and are located



*Fig. 219. Roof brick arrangement recommended by Electric Furnace Refractories Sub-Committee.*

at the corners of an equilateral triangle. They are raised and lowered pneumatically by electric or hydraulic gear.

In most modern installations the bulk of the charge is introduced to the furnace by means of a "basket" or skip (fig. 217, p. 511) temporarily located above the furnace body—the roof having been previously removed. Such rapid charging is a great advantage of the arc process, particularly as the heat from the electrodes is soon released within the charge due to their deep penetration. Any additional scrap or feed is introduced through the side doors, of which there are generally one or two. The general operating procedure for the basic process has been well summarised in the A.S.T.M. Standards on refractories: the charge is melted down under an oxidising slag, to remove phosphorus and also

carbon, silicon, and manganese, as desired. Usually it consists of steel scrap, alloys and alloyed scrap, and often a small amount of limestone. After charging intermediate voltages are first applied to enable the electrodes to bore without excessive radiation to the roof. Later a high voltage is used in order to decrease oxidation losses. When the scrap is part melted the first slag additions are made, these usually consisting of lime with a small amount of fluorspar, or sand, as a flux. The heat is normally finished by one of three slag practices:

- (1) Under the original oxidising slag.
- (2) By adding ferrosilicon to make the original oxidising slag reducing.
- (3) A two-slag process—the most commonly used—in which the oxidising slag is removed by tilting the furnace backwards and removing the slag through the charging door. A reducing slag, consisting of lime, silica, fluorspar, coke or ferrosilicon, is then added and the heat finished under this powerfully desulphurising and deoxidising cover.

The slag so formed is often referred to as a falling slag, since on cooling to room temperature, the dicalcium silicate contained therein changes from the beta to the gamma form with a volume increase of 10 per cent. Where this change occurs in slag absorbed in the hearth it may well lead to localised damage.

The melting and refining times vary considerably with the size of furnace and the process operated, but for a 10-ton furnace 2 hours to melt and 3½ to refine may be taken as typical, or 3 hours to melt and 4 hours to refine on a 60-ton furnace. In addition fettling normally occupies approximately 45 minutes.

The general conditions existing in the arc furnace are summarised in Table XLVIII, which is based on one given in the above mentioned A.S.T.M. manual, whilst fig. 218 gives a typical arrangement of refractories used. It will be seen that the construction is very much simpler than that of the open-hearth furnace, lending itself to discussion under 3 major sub-divisions, *viz.*, the Roof, Side Walls and Bottom.

## THE ROOF

### (Section 2)

Detailed consideration was given in the latter part of the Second World War by the Electric Furnace Refractories Sub-Committee of the Open-Hearth Refractories Joint Panel to the factors controlling arc furnace life, data being collected from a large number of British firms, on both flat centred and domed roofs of the fixed and movable type.

TABLE XLVIII  
CLASSIFICATION OF REFRACTORIES FOR ELECTRIC-ARC STEEL FURNACES (A.S.T.M.)

Furnace part	Material		Temperature* °F. (°C.)	Slagging action	Abrasion	Load	Spalling	Remarks
	Basic type furnace	Acid type furnace						
Hearth bottom and banks	Magnesite types of grain or ramming mixtures, dolomite	Silica sand or ganister grain	2650 to 3100 F. (1455 to 1705 C.)	Severe	Severe	Unimportant	No	Infiltration of slag or metal into cracks may cause disruption of bottom
Side walls	Silica brick; magnesite brick; burned, chemically bonded, or metal encased	Silica brick	2650 to 3100 F. (1455 to 1705 C.)	Moderate	Mechanical abrasion of charge may be severe	Unimportant	Severe	Tilting of furnace may cause shifting of walls
Roof	Silica, kyanite, or alumina-diaspore brick. Kyanite or mullite ramming mixtures	Same as for basic type furnace	2650† to 3100 F. (1455 to 1705 C.)	Severe around electrode holes	Unimportant	Arch stresses	Severe	
Tap hole	Clay tile, basic brick	Clay tile	2600 to 3100 F. (1425 to 1705 C.)	Severe	Unimportant	No	Severe	Erosion is severe
Spout	Clay or basic brick, plastic chrome or magnesite mixes, loam, fire clay	Clay brick fireclay, loam	2600 to 3100 F. (1425 to 1705 C.)	Severe	Unimportant	No	Severe	Erosion is severe

\* The equivalent Centigrade temperatures given in parenthesis are to the nearest 5 degrees.

† Direct fusion may occur due to high temperature of electric arc.

As a result of this detailed investigation, certain recommendations were made regarding roof design, which although not universally adopted have proved quite satisfactory and do not require the use of the absurdly large number of shapes previously employed.

#### (a) CONSTRUCTION

##### *Roof contour*

The Sub-Committee's studies having suggested that flat centred roofs, in addition to demanding special shapes, were more subject to spalling, it was decided to standardise the domed type, as shown in fig. 219. Such roofs are widely used and the roofs of most arc furnaces could be built with this general design.

##### *Thickness*

There has been a tendency in the past to make large roofs thicker than small roofs but it was considered that all furnaces in the diameter range 5 ft. to 15 ft. could be satisfactorily equipped with 12 in. roofs.

##### *Rise*

Typical practice is the use of 1 in. per ft. of diameter, which is rather less than that employed on open-hearth furnaces.

##### *Size of bricks*

It is now generally known that large bricks are more subject to spalling than small bricks, even though they are made of material of identical properties. Furthermore small bricks can be manufactured with greater accuracy and fewer making faults. They also permit of more uniform distribution of expansion joints and are, therefore, strongly recommended.

##### *Areas between electrode rings*

This region is frequently a difficult one, particularly since variations, due for example to distortion in the skewback ring, must be accommodated. At one time large plugs were standard in this region but there would appear to be a good case for using a similar bricking in this part of the roof to that employed in the remainder.

The crown of an arch or dome is referred to as a key brick and must be accurately made to conform with the general roof contour.

##### *Electrode rings*

It is recommended that these be built of side arch bricks of the correct taper, their length being such as to cause protrusion from the

upper surface of the crown and allow of the coolers occupying a horizontal position. Protrusion of electrode bricks below the general roof surface not only lacks advantage but is considered dangerous, since spalling of the projecting ends may well cause fracture behind the general face of the roof.

#### *Channel or skewback ring, or skewback bricks*

Most modern furnaces are fitted with water-cooled skewback rings, often with a "built-in" slope, obviating the necessity of using special skewback bricks. Older furnaces either with uncooled or inadequately cooled rings frequently show distortion and, therefore, require frequent checking if deterioration of roof life due to unequal stress distribution is to be avoided.

Where special skewback bricks are employed they should not be excessively large, the width from front to back being limited to a maximum of 12 in. and the thickest part not being greater than  $3\frac{1}{2}$  in. Any rebates necessary to accommodate channel plates should be radiused as much as possible in order to reduce cracking in manufacture.

#### *Building*

It is recommended that all roofs be built circumferentially, the peripheral dimensions of the bricks (inner and outer edges) being so arranged that the inner edges of the bricks in any one course should be less than the outer edge comprising the next inner course. The object of this is to safeguard against the bricks of the inner course becoming displaced.

#### *Expansion allowance*

As with other silica constructions some expansion allowance is desirable, the use of tarred roofing paper providing a satisfactory method. The actual amount of expansion allowance varies considerably with individual practice but a figure of  $\frac{1}{8}$  in. per ft., which is in general line with the known expansion of silica (about 1.25 per cent. to 1000°C.) is considered reasonable.

#### *Brick types*

If the above principles are followed then roofs between 5 ft. and 15 ft. in diameter can be divided into three groups, *viz.*, 12 ft. to 15 ft., 8 ft. to 12 ft., and 5 ft. to 8 ft., each of which can be constructed with a high degree of accuracy using 1 size of centre brick, 1 size skewback, 6 sizes of arch brick and 1 size side arch brick, for the electrode ring. Details of the actual sizes are given in the Iron and Steel Institute Special Report No. 32, together with a schedule of proportions and



quantities required. It will be seen from this that some rings are built from bricks of a single size, whilst others are built of bricks of 2 sizes used in specified proportions.

There are of course numerous other methods of constructing arc furnace roofs, ranging from such modifications as the use of ribs either of silica or sillimanite, to the complete replacement of the silica by a monolithic structure. The proportion of roofs employing such unconventional features is, however, small.

#### (b) MATERIALS

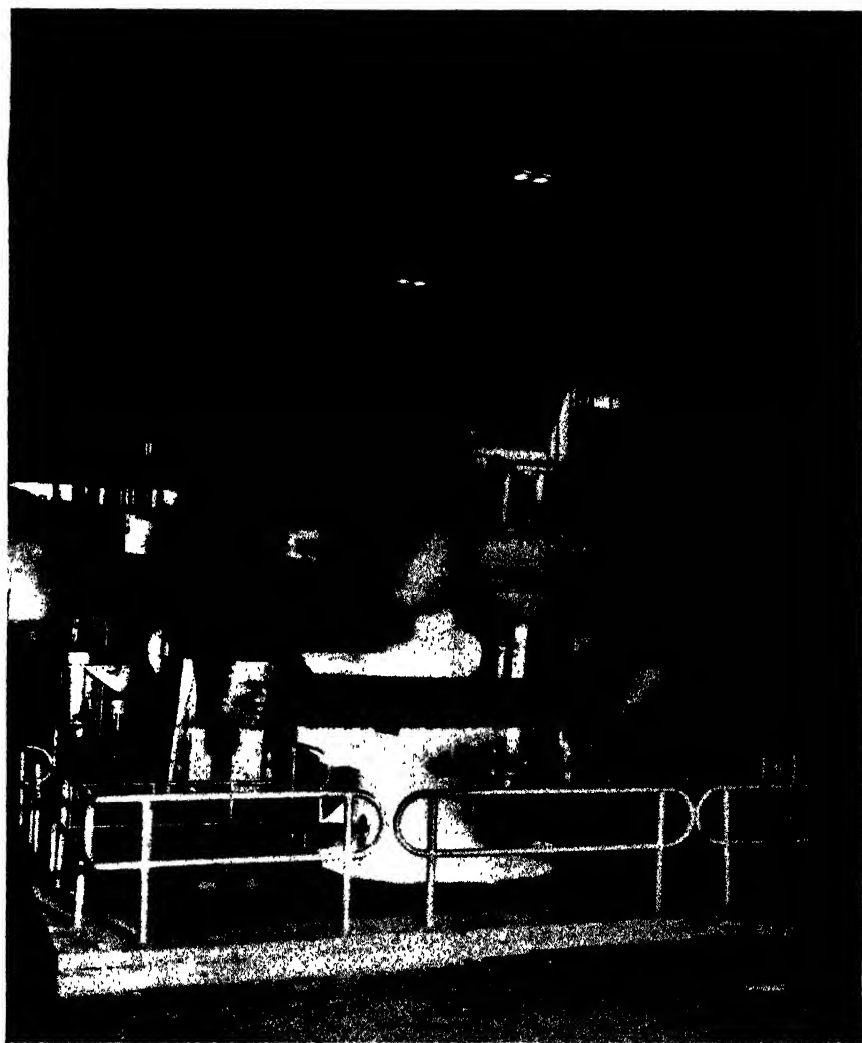
Silica is by far the most commonly used material for arc furnace roofs, and it may be taken that the properties required in such bricks are essentially those specified for first-quality open hearth furnace roof bricks (*see* Chapter VIII). Many firms however replace the silica bricks in the outer 2 or 3 courses by fireclay bricks of the 42 per cent. alumina type. The advantage of this procedure is that the spalling frequently observed in these outer rings (*see* fig. 220, p. 512) is substantially reduced, while the slag resistance although not as high as that of silica, is found to be adequate for this relatively cool position. Hütter even suggests the use of special magnesite bricks in this position and claims that their use reduces the damage normally caused to the top of the side walls by silica drip.

For special purposes, and in particular with small furnaces used periodically and, therefore, subject to a great deal of thermal shock, better results are sometimes obtained by replacing all the silica by, say, super-duty fireclay brick of 45 per cent. alumina content, or even sillimanite (63 per cent. alumina) or other high alumina refractories. Super-duty fireclay bricks are characterised by good thermal shock resistance (compared with silica) and do not shrink as readily as the ordinary fireclay qualities. Although their pyrometric cone equivalent is roughly 60°C. higher than that of first-quality silica, they are if anything more heavily attacked by ferrous oxide. Sillimanite is physically extremely satisfactory, in that it has high refractoriness, freedom from shrinkage, and a high refractoriness-under-load. Unfortunately its price is many times that of silica, whilst its resistance to iron oxide is much the same.

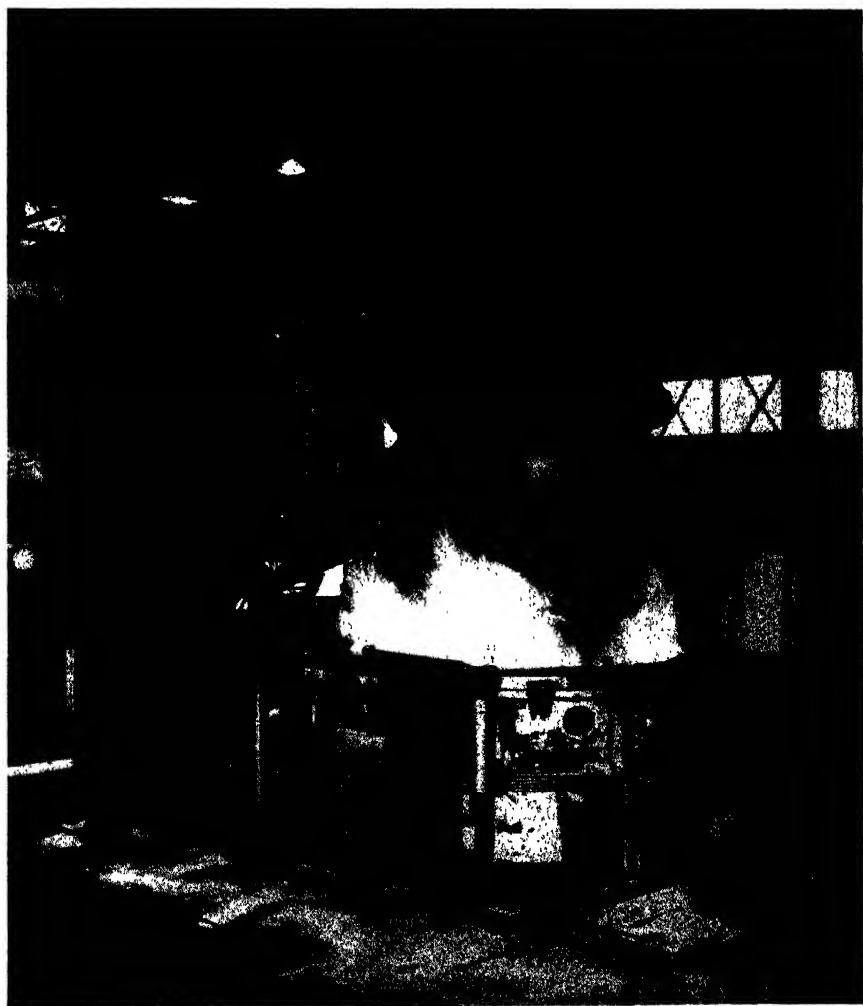
Many trials have been made of other bricks, *e.g.*, magnesite, chrome-magnesite, and forsterite, but the results obtained do not appear in general to warrant the far higher initial cost, except perhaps for certain special purposes, *e.g.*, the manufacture of ferro-alloys. Where monolithic roofs have been made, usually on small furnaces, the refractories



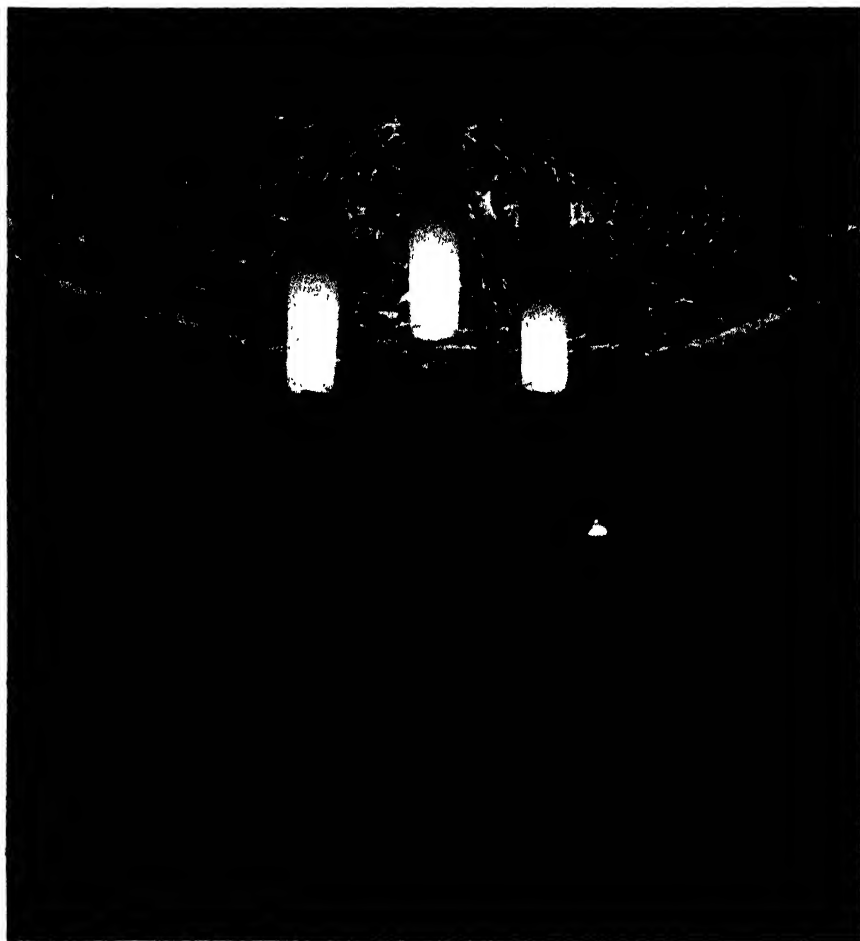
*Fig. 215. 60-ton arc furnace.*



*Fig. 216. 60-ton arc furnace tapping into ladle.*



*Fig. 217. 60-ton arc furnace showing body displaced from roof and basket charger.*



*Fig. 220. Arc furnace roof showing spalled areas adjacent to skewback channel.*

tried are usually those of the kyanite or mullite type, but also include such radically different materials as doloma.

(c) *Life and causes of failure*

If arc furnace roofs were as difficult to change as open-hearth furnace roofs, operators would doubtless be more concerned about the short and variable lives at present obtained. In some plants even a life of 30 to 40 heats is considered quite good, but at others lives of over 200 heats are obtained. Many factors are responsible for these differences, for example the type of steel being made, the production rates and above all the frequency of use, small furnaces used periodically being subject to severe spalling. One factor having a marked effect on roof life is the height of the roof above the bath. Merely raising the roof would be sufficient to increase the life in many plants, though doubtless at the expense of greater power consumption. In view of the known sensitivity of silica bricks to spalling when heated rapidly to red heat, and the inevitability of a quick heat-up when a new roof is placed on an already hot furnace, it might be expected that the preheating of roofs, say with gas, would be standard practice. In point of fact it is rarely employed, and in consequence an appreciable amount of spalling is considered inevitable (*see* fig. 220, opposite).

M. P. Fedock, in his discussion of factors affecting refractories performance in arc furnaces, suggests that damage due to high temperatures and to spalling have been over-emphasised, and that "It's not the heat but the fumidity that makes the refractory uncomfortable!". By examining the working face of a number of used roof bricks of known history and by taking samples both of furnace fumes and of the gas atmospheres, he arrives at a number of extremely interesting and novel conclusions. As will be seen from Table XLIX a variety of oxides are absorbed by the furnace roof, the amounts of  $MnO$ ,  $Cr_2O_3$ ,  $CaO$  and particularly  $MgO$ , present on the working surfaces, being surprisingly large, particularly when compared with those found in open-hearth furnaces. Fedock shows that the composition both of the fumes and the gases is characteristic of the melting practice, being due to differences in the composition of the materials charged and the slags formed. Thus he shows that with an oxygen-blown carbide-slag practice, the fumes on melt down are essentially gamma hematite, but that during the oxygen blow much denser fumes, containing, in addition, magnetite, are produced. After slag-off, when the carbide slag is formed, the fumes are chiefly lime and magnesia with appreciable amounts of silica and fluorspar. In stainless melting practice on the other hand, the fume produced early in the melt was found to consist chiefly of chromite

**TABLE XLIX**  
**TYPICAL WORKING-FACE ANALYSIS, SILICA ROOF BRICK FOR ELECTRIC FURNACES**  
 (After Fedock)

<i>Furnace practice</i>	<i>Chemical analysis, Pct</i>							<i>Mineral phases other than those expected from fused silica brick</i>
	<i>SiO<sub>2</sub></i>	<i>Al<sub>2</sub>O<sub>3</sub></i>	<i>CaO</i>	<i>FeO</i>	<i>Fe<sub>2</sub>O<sub>3</sub></i>	<i>MnO</i>	<i>Cr<sub>2</sub>O<sub>3</sub></i>	<i>MgO</i>
Stainless, not oxygen-blown	92.88 89.80	0.78 1.49	2.24 2.24	0.21 0.21	1.36 4.24	0.18 0.18	0.40 0.00	0.20 0.16
Stainless, oxygen-blown	88.70	1.20	2.46	1.82	1.20		3.63	0.36
High-carbon alloy,* carbide slag:								
$\frac{3}{8}$ in. face	90.50	0.33	3.34	0.36	2.00	0.36	0.00	2.35
$\frac{1}{2}$ in. face	90.16	0.50	2.26	0.29	1.92	0.47	0.00	3.14
$\frac{1}{16}$ in. face	62.88	0.68	3.40	0.86	13.12	3.51	0.00	14.63
Low-carbon alloy,* carbide slag	88.76	0.29	3.23	0.56	1.62	0.20	0.31	3.61
Oxygen-blown alloy,* carbide slag	88.60	0.27	4.66	0.96	1.13	0.71	0.19	3.44
Single slag	86.70	1.10	2.65	0.72	9.71	0.11	0.00	0.16
Unused silica brick	95.0	0.85	2.12		1.72		0.00	0.30

\* Usually metallic pellets could be observed embedded in the working face of these bricks. The pellets ranged in size from  $\frac{1}{8}$  in. down.

spinel ( $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ ) and magnetite ( $\text{Fe}_3\text{O}_4$ ), whilst during the oxygen blow, the fume was chiefly a refractory chromite spinel. Fedock goes as far as to suggest that for Canton practice, the relative corrosiveness of different melting practices, can be assessed (*see* Table L). It will be seen from this that on a 15-ton furnace the wear on the roof is more than 6 times as great for oxygen-blown alloy steel as for oxygen-blown stainless. He suggests that the refractory chromite spinels produced in copious amounts during oxygen blowing actually shield the roof from bath radiation, and also coat the working surface of the roof without having any serious fluxing action.

TABLE L  
RELATIVE CORROSIVENESS\* OF MELTING PRACTICES ON  
FURNACE REFRACTORIES  
(After Fedock)

<i>Melting practice</i>	<i>Silica roof</i>		<i>Metal-encased magnesite-chrome side walls</i>	
	<i>15-ton furnace†</i>	<i>15-ton furnace†</i>	<i>15-ton furnace†</i>	<i>50-ton furnace†</i>
Oxygen-blown alloy ..	6.45	5.50	3.96	
High-carbon alloy ..	2.70	3.20	2.56	1.95
Low-carbon alloy .. ..		2.90		1.95
Single slag .. ..		1.55		1.65
Oxygen-blown stainless ..	1.00	1.00	1.00	1.00

\* Factors were calculated relative to oxygen-blown stainless practice, which is least corrosive and taken as unity.

† Performances on three 15-ton furnaces and six 50-ton furnaces used to calculate the factors.

The maturing and zoning observed with open-hearth furnace roofs is by no means as marked with arc furnace bricks, possibly because the surface layers melt away before they have time to mature (the average life being much shorter than in the open-hearth) or because of the strongly reducing atmosphere. The most surprising difference between used open-hearth and arc furnace roof bricks is, however, the predominance of tridymite in the working face of the arc furnace brick instead of the cristobalite found in all open-hearth roofs. It was at one time thought that this might be due to relatively low operating temperatures, the excessive wear being caused by reducing conditions. More



recent results, however, suggest that it is the state of oxidation of the iron which determines the final form of the silica, and that where ferrous oxide is present tridymite forms rapidly once the temperature falls below  $1470^{\circ}\text{C}$ . That conditions may be strongly reducing was well brought out by Fedock, who found as much as 93.8 per cent. of carbon monoxide in the furnace atmosphere on a high carbon alloy steel, 1 hour before tapping, with zero carbon dioxide and only 1.6 per cent. oxygen. The same sample contained 3.6 per cent. of hydrogen, and only 1 per cent. of nitrogen. Like most gas samples it is probably a mixture of smaller increments and not, therefore, in equilibrium, but the overall analysis certainly suggests strongly reducing conditions. Incidentally very few of Fedock's gas analyses show more than 2 per cent. oxygen, though one taken towards the end of a stainless melt showed over 12 per cent.

In many plants roofs come to a premature end because of a local hot spot, frequently in the region of the electrode rings, where the escape of hot dirty gases may well lead to high temperatures, high flux deposition rates and physical erosion. Given the right technique new electrode rings can be inserted in most roofs, and by doing this 2 or 3 times in a campaign the life obtained may well be doubled.

#### (d) LINES OF IMPROVEMENT

Most plants could obtain a useful improvement in roof life given sufficient attention to detail, for example, maintenance of roof ring shape, correct brick size and expansion allowances, and improved repair techniques. Where spalling of the outer rings is still a problem, the use of spalling resistant bricks in these positions may also prove advantageous. Three lines of development are suggested as at present worthy of closer attention, *viz.*:

- (1) Controlled preheating of new roofs to such a temperature as to enable them to be transferred to the furnace without falling below  $300^{\circ}\text{C}$ . or preferably  $600^{\circ}\text{C}$ .
- (2) The use of silica bricks of unusually low flux content and porosity, such as are now giving greatly improved performance on open-hearth furnaces.
- (3) The introduction of fume control: an admittedly vague suggestion based on the stimulating information supplied by Fedock, and parallel experience in the open-hearth furnace.

Studies in open-hearth furnaces have already shown that the formation of iron oxide and, therefore, the attack on roofs, is influenced by such factors as bath conditions, the type of fuel used and steam/oil ratio. It



*Fig. 221. 60-ton arc furnace showing use of magnesite-tubes in side walls.*



is by no means impossible that similar factors might be discovered for the arc furnace and that quite practicable changes, either in charging practice or slag control, might have an appreciable influence on roof life. As with the open-hearth furnaces, the real need is for a cheap basic roof that would not only be capable of a far higher temperature but have a much greater resistance to basic fluxes. Such work as has been done in this field, however, suggests that structural spalling of the type found in the open-hearth is likely to be the main difficulty. Furthermore the use of refractories containing chrome is often dangerous, since brick ends falling into the slag may rapidly lead to chromium in the charge under the strongly reducing conditions frequently present.

## SIDE WALLS

### (Section 2)

#### (a) CONSTRUCTION

Most modern furnaces are lined either with simple brick shapes or tubes but considerable development is still taking place in the field of monolithic linings and this may well lead eventually to a change in practice. At present monolithic linings made from tarred doloma are mostly employed in plants where the raw material is already being prepared for other purposes, *e.g.*, basic bessemer converters. An intermediate procedure, *viz.*, the production of large preformed segments, which can be lowered into the furnace and which permit of a more rapid relining, is now being tried in some plants. Thus Müller describes the application in East Germany of a Soviet method of using segments made from doloma and bitumen. These are jointed in the furnace with a slurry consisting of magnesite and water glass. Where brick or tube linings are employed they are usually 14 in. to 18 in. thick and have a vertical working face. They are built in numerous ways, *e.g.* with a 9 in. basic working face and two soldier courses between this and the case, or with a 14 in. inner wall of tubes with one soldier course behind it (*see* fig. 221 p. 517). Expansion allowances may be left either as vertical joints in the brick lining, as crushable insulation, or as an air gap between the refractory lining and the steel jacket. Bricks, particularly if metal-cased, may be set dry, but where tubes are employed the gaps between must be filled with magnesite cement, normally of relatively low moisture content.

#### (b) MATERIALS

Many furnaces still employ silica bricks, at least in the top courses of the side walls, though the development of improved types of basic

brick are leading to their extended use. One of the most useful types of basic brick is that originated by McCallum in 1914, in which lengths of scrap tube are filled with ground magnesite. The more recent development of the steel jacketted standard shape is, however, proving a strong competitor, particularly since it permits of more rapid laying. Such bricks are made by introducing into a hydraulic press box a "U"-shaped steel liner fitted with lugs. The magnesite is poured into the mould in the ordinary way, and the brick pressed, the top side of the steelbox being formed by a sheet of metal attached magnetically to the top plunger. An additional advantage of such bricks is that the magnesite can be pressed under an extremely high load, whereas the McCallum type are usually hand or pneumatically rammed. This latter type can be obtained with a straight magnesite filling, but may contain an addition of approximately 20 per cent. of chrome ore, which is found to improve volume stability and reduce the shrinkage observed with straight magnesite at high temperatures.

Many other types of brick have given satisfactory service in arc furnace side walls, *e.g.*, standard chrome magnesite or semi-stable dolomite bricks. The life obtained with these in 12-ton furnaces has been found to be of the order of 4 weeks compared with 12 or 13 weeks for magnesite tube linings. In older furnaces the charging arch was normally built of silica, other bricks showing insufficient refractoriness-under-load. In most modern furnaces, however, no such arch is required, this section being replaced by a water-cooled head-plate. For the tapping arch magnesite bricks are preferred, the use of chrome magnesite although reducing spalling difficulties being dangerous due to the risk of chromium pick-up in the steel during tapping.

#### (c) LIFE AND CAUSES OF FAILURE

Here again the life obtained varies greatly from one plant to another, being as low as 2 or 3 weeks or as long as 13. The service conditions are far from easy, since in addition to high temperatures, excessive fume and substantial thermal shock, the lining is subject to heavy abrasion. It is not difficult to imagine that basket charging although in general an admirable procedure is likely to result in the immediate removal of any spalled or loose brick ends.

In some plants linings wear back fairly evenly but in others it is usual for shrinkage to occur, resulting in overhanging sections and localised collapse. At one time such local failures always necessitated the furnace being taken out of production, but more recently effective repairs have been achieved by throwing a suitable basic cement into the affected part by means of a compressed air gun.

#### (d) LINES OF IMPROVEMENT

Since roofs can be readily changed, and a furnace bottom may be expected to last about a year, the side wall is really the bottleneck in arc furnace production. The improvement of bricks for use in this position, and the development of more rapid methods of installing linings, would therefore justify a good deal of attention. It may well be that furnace availability will eventually be increased by the use of preformed sections, such as are now already being tried. These latter are at present of the monolithic type, but trials have been made in open-hearth furnaces of prefabricated brick panels and there is little doubt that similar techniques could be developed for use in arc furnaces. An obvious method for increasing stability is the use of a truncated cone section rather than a cylindrical lining. This would provide the effect obtained in the open-hearth furnace by the battering of back and front walls, but would if applied to existing furnaces result either in reduced capacity for scrap or in an undesirably thin section at the top of the lining.

### THE HEARTH AND BANKS

#### (Section 3)

##### *The sub-hearth*

Certain of the more recent hearth installations, *e.g.*, those employing the patented Crespi process, are said to be made without the use of any sub-hearth, *i.e.*, the graded doloma is rammed on the pan itself and only sintered in position by the heat of the steelmaking process. Most furnaces still contain a sub-hearth, which generally consists of 9 in. to 12 in. of prefired brick. A large number of combinations are used, as examples may be given a furnace with a 3 in. high temperature insulating brick on the pan with 3 in. firebrick and two 3-in. courses of magnesite above; a second in which there is no insulation and in which the 3 in. firebrick are covered with three 3-in. layers of magnesite; and the third in which a dry rammed dolomite lining is supported on two 3-in. courses stabilised dolomite brick raised on one 3-in. course of firebrick. The bricks are arranged either in parallel strips across the furnace or in a series of rings starting from the outside and working towards the centre. In both types the bricks are arranged so that there are no straight through joints through which slag or metal can penetrate. In some installations chrome bricks or chrome plastic are used to separate the firebricks from the basic brick hearth above, but the use of such a neutral course is not necessitated by the normal temperature conditions. Certain operators are reluctant to consider the use of

stabilised dolomite bricks in the sub-hearth, particularly where a break-out might cause damage to ancillary equipment. Long term experience however suggests that breakouts are no more likely to occur through stabilised dolomite sub-hearths than through hearths made of straight magnesite bricks.

#### THE MONOLITHIC HEARTH

At one time most British arc furnace bottoms were rammed in with tarred doloma, whilst most American furnaces were equipped with magnesite bottoms burned in successive layers, or rammed with a suitable bond. The extra demands placed on arc furnaces using oxygen lancing in the production of low carbon stainless steel have necessitated the use of magnesite bottoms in certain British furnaces but there has also been a marked tendency to go over to dry rammed doloma hearths of the Crespi or Swift type. The main motives behind this latter development are the reduction in installation time and the relative imperviousness of such hearths to metal. Where tarred doloma is employed both the raw material itself and the installation procedure are essentially similar to those already described in Chapter IX on the basic open-hearth furnace. There is, however, a difference in burning in, since the heat has to be generated by means of electricity rather than liquid or gaseous fuel. This is normally done by placing scrap electrodes in a "T" formation on the bottom, which act as resistors when the electrodes are lowered. With dry rammed doloma hearths on the other hand the bottom is covered with steel plates, the scrap charged in the ordinary way and a heat carried out without any preliminary burning in.

Magnesite hearths were at one time also burned in layer by layer, but the more modern practice makes use of proprietary ramming materials containing a bond that permits of their being completely rammed before heat is applied. The materials most commonly used can be divided into two classes: those containing about 65 per cent.  $MgO$  and those containing about 93 per cent.  $MgO$ . The former contain substantial quantities of lime and silica and are normally bonded with sodium silicate. The latter are essentially sea-water magnesia with a soluble magnesium chromate bond. Both types have a grading consisting of approximately 60 per cent. of 28 (Tyler) mesh and 35 per cent. through 100 mesh. These materials are mixed with a small amount of water and are pneumatically rammed in a series of 3 in. layers, which are vented with a nailed board or a drill to facilitate drying.

Where the main product is stainless or other high chromium steels the hearth may be built of chrome bricks and repaired with chrome ore.

Such hearths are said to be satisfactory even at temperatures around  $1800^{\circ}\text{C}$ .

The life of the bottoms varies considerably from one plant to another but a reasonable life on a dolomite bottom of a 10-ton furnace is about 1 year, or 1000 casts. It should be remembered, however, that very little of the original bottom remains after such a campaign, the amount of doloma used in the initial installation being very small compared with that employed for fettling. The actual fettling figure varies with steel quality and material charged, but for doloma is of the order of 25 lb. to 50 lb. per ton of steel made. Longer lives can be obtained by careful fettling, but arc furnace bottoms, like those in the open-hearth, tend to get out of condition due to metal penetration, with a consequent increase both in "bad bottom" time and breakout risk. Since the installation of a new bottom is comparatively simple and the cost small when spread over the life, it is better to renew a bottom regularly than to go for a record that might in the end prove far from economic. Incidentally the formation of a weak zone in an arc furnace bottom can be clearly shown by taking periodic readings of a series of thermocouples introduced a short distance into the hearth through holes in the bottom. Fig. 222, shows the gradual development of a weakness, whilst later studies showed that given suitable fettling the original temperature distribution could be restored.

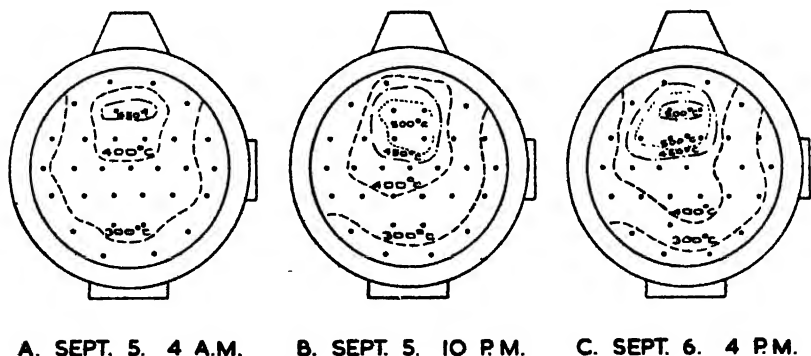


Fig. 222. Temperature contours 3 in. above the pan in 60-ton arc furnace bottom showing gradual development of hot spot (R. H. Baulk).

## DOOR AND LANDER

The refractories used in the doors and landers of arc furnaces are essentially similar to those employed in the open-hearth furnace, *i.e.*, the doors are usually lined with a 35 to 38 per cent. alumina firebrick



having a melting point of about 1700°C. and a good thermal shock resistance, while the lander is lined with similar brick, parged with "ganister", *i.e.*, clay-bonded quartzite. This latter is also prepared in the manner described in Chapter IX on the open-hearth furnace.

## THE ACID ARC FURNACE

For certain purposes, in particular the manufacture of steel castings, materials low in phosphorus and sulphur are charged on to an acid hearth and a highly siliceous type of slag employed. The conditions to which the refractories are subjected are less severe than in the basic process, and the general arrangement of refractories, which is usually simple, has been fairly fully described by Demler and Pierce.

## THE ROOF

### *(Section 1)*

This is in every way similar to that employed on a basic open-hearth or arc furnace.

## SIDE WALLS

### *(Section 2)*

These are built of silica brick, often laid dry with the normal  $\frac{1}{4}$  in. per foot expansion allowance. Where necessary super-duty silica refractories can be employed.

The door arches and jambs are also normally built of silica, though high alumina refractories are sometimes used in order to minimise spalling.

## THE HEARTH AND BANKS

### *(Section 3)*

The sub-hearth consists of silica bricks, often laid on 1 or 2 courses of fireclay brick. Silica bricks are laid dry and there is the usual expansion allowance, which is often confined to an annular space under the side walls next to the shell. The monolithic bottom usually consists of a mixture of ganister and plastic fireclay, the ganister varying in size from  $\frac{3}{8}$  in. downwards and the fireclay varying from 10 to 20 per cent. This mixture is either sintered into place in layers, by means of the arc, or completely rammed pneumatically, each layer being roughened before the next is applied. The water content is kept as low as is consistent with good ramming qualities. Bottoms of this type frequently last as much as 2000 heats.

## INDUCTION FURNACES

The coreless type of induction furnace, which has largely replaced the old crucible process, is essentially an air core transformer in which the furnace coil is the primary and the charge the secondary. The furnace is very simple (*see* figs. 223 and 224, pp. 527 and 528, and fig. 225 below)

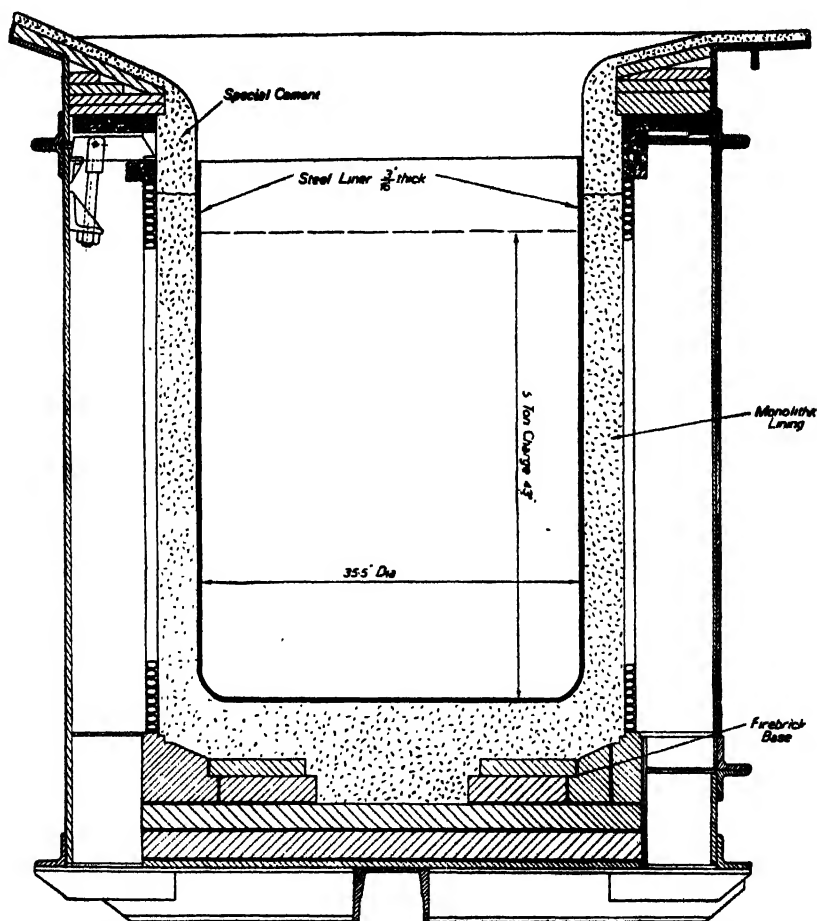


Fig. 225. Use of steel liner for forming the lining of a 5-ton acid induction furnace.

consisting essentially of a water-cooled copper coil through which is passed a high-frequency alternating current. Given the correct coupling between coil and charge, the eddy currents produced in the latter cause it to heat up and eventually to melt. Small furnaces for melting fine

materials normally operate with extremely high frequencies but with medium capacities of the order of 1 ton the frequency falls to about 500 cycles, and for still larger furnaces the ordinary mains frequency (50 cycles per second) can be employed. Although in the early days furnaces of even 1-ton capacity were considered remarkable, furnaces of 2 to 5 tons are now fairly common, whilst in Sweden several furnaces have been built capable of tapping 12 tons.

The coreless induction furnace differs radically from the arc furnace, in that the composition of the product is almost entirely controlled by the composition of the charge, and the minor losses which occur during melt down. Given sufficient experience these latter can be predicted and, in consequence, steel made to close specifications. Attempts to use refining slags have mostly failed due to the lack of top temperature. Such furnaces are generally employed for the production of special high grade steels, such as stainless, extremely low carbon alloy steels, high manganese steels and tools steels.

Before the present coreless type came into its own, large furnaces were constructed, for example the Kjellin and Röchling-Rodenhauser, in which the charge was heated in a ring-shaped trough and use made of an iron core to facilitate the coupling. Some of the early types were lined with silica bricks, but these did not last particularly well and tended to result in silicon pick-up by the charge. Later furnaces were lined with dead-burned or electrically-fused magnesia and also with tarred doloma.

Magnesite linings made in this way were stated to show no signs of softening at 2000°C. and gave a metallic ring when struck at the working temperature. They averaged about 400 heats when melting steel and it is stated that no patching was necessary for the first 100 heats.

#### PROCEDURE USED FOR LINING CORELESS TYPE FURNACES

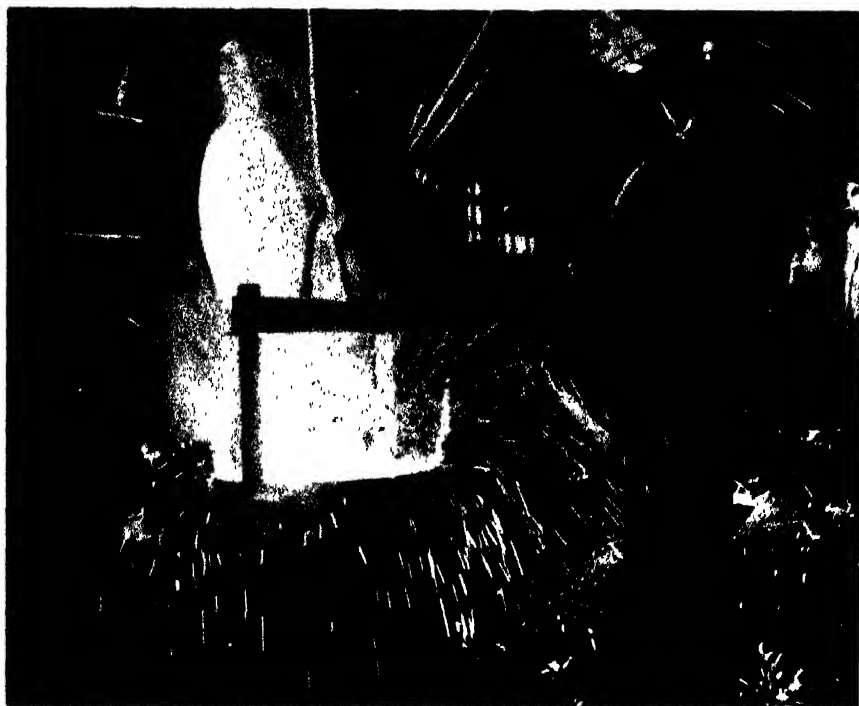
In the early furnaces the charge was normally melted in a crucible. As, however, the size of furnace increased, the production of a crucible that would give a reasonable life became increasingly difficult. The different methods used in lining induction furnaces are summarised below, Method 2, in which a steel liner is employed, being virtually standard in Great Britain.

##### 1. *Pre-formed crucibles*

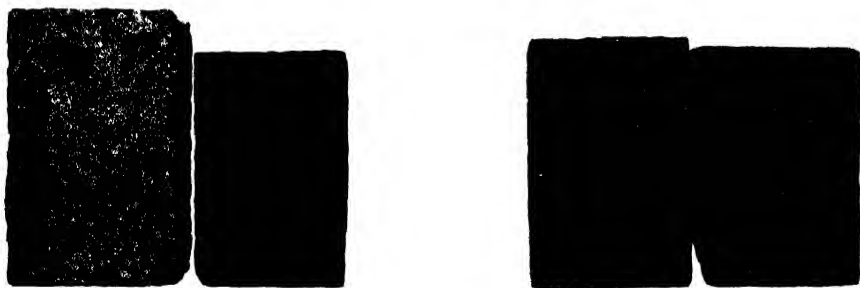
The first crucibles were usually made either of fireclay or plumbago (clay-graphite), the former giving a life of 4 or 5 heats and the latter lasting somewhat longer. Other materials were employed, e.g., magnesite



*Fig. 223. Induction furnace section of an electric steelplant, showing 5-ton acid furnace tapping into ladle.*



*Fig. 224. Close-up of 5-ton acid induction furnace tapping into ladle.*



*Fig. 229. Expansion or contraction of induction furnace linings (test-pieces originally same size) after firing for 1-hour at 1600°C. Left to right: Acid lining, basic lining and 30-70, 50-50 and 70-30 magnesia-alumina mixtures.*

and magnesia-zircon, but were found susceptible to thermal shock. Attempts were made to overcome this difficulty by lining fireclay or graphite crucibles with various cements, for example Northrup made use of magnesia and zirconia base cements in graphite crucibles. Such linings have, however, been found to have very limited applications, partly because they wear away quite rapidly, but also because cracking may cause contamination due to carbon pick-up from the plumbago crucible.

2. *Monolithic linings—the steel liner method*

This procedure, as initially developed by Rohn, who patented the use of a dry granular material sintered in position by the melting of a steel liner, has the advantage that it can be used over a wide range of furnace size (*see below*).

3. *Monolithic lining—the asbestos liner method*

Considerable success has been obtained, particularly in the United States, by ramming the granular refractory between the coil and an asbestos cylinder. The furnace is charged in the ordinary way, the reaction between the asbestos and the refractory lining during melt-down being sufficient to give adequate sintering.

4. *Brick linings*

The bricks may be fired or unfired and made from acid or basic materials. They usually take the form of curved tiles with tongued and grooved edges.

INSTALLATION OF LININGS USING A STEEL TEMPLATE

(*Method 2*)

(a) *Construction*

The procedure normally employed in installing acid or basic linings in British high-frequency furnaces can be summarised as follows:

The bottom of the coil (*see fig. 225*) is first bricked to give a "sand-tight" seal. With basic linings magnesite or fireclay brick are employed and with acid linings silica or fireclay brick. Approximately 6 in. of lining material is then poured on to the top of this brickwork, smoothed out, and rammed in position using a tool with a fairly large head, say about 2 in. in diameter. The bottom is further consolidated by means of a chisel-edge tool. To the spectator the use of such a tool for ramming may appear absurd, but early experience soon showed that sharp edged tools were far more effective in compacting granular linings than blunt ones and indeed that the exclusive use of blunt tools

might well result in pockets in the lining and consequently in a serious risk of breakout. It is interesting to note that in the dry rammed, *e.g.*, Crespi type arc furnace or open-hearth furnace bottom, the ramming is also done with sharp ended tools.

The next step is to place the steel liner, which consists of a cylinder closed at the lower end by a domed plate, on the material first rammed and bed it in position. Four wooden spacers are then placed vertically between the coil and the steel liner in order to ensure constant lining thickness in the subsequent side-wall ramming. This latter is done by pouring the powder into the gap between the coil and the steel liner and ramming in position in layers of 2 in. to 3 in. depth. After each layer has been rammed, the wooden spacers are lifted and care taken to see that no weak spots develop in these four positions. When the lining has been rammed to within a few inches of the top, it is finished off by using a cement such as can conveniently be made by mixing a quantity of lining with a water-glass solution. In small furnaces, the top of the lining is sometimes protected by the use of a heavy iron ring—usually about  $\frac{1}{8}$  in. thick, the ramming being continued between this ring and the furnace coil.

When the furnace is required for production, scrap is charged and the power switched on. The template first becomes red hot and after being maintained at a temperature of the order of 1300°C. for an hour or so it splits from top to bottom by melting and more power can be employed. The charge then starts to heat up rapidly and can be tapped in 1 or 2 hours in furnaces ranging from  $\frac{1}{4}$  to 2-ton capacity.

#### (b) MATERIALS

##### *Basic refractories—magnesia type*

In the early and rather frightening days of induction furnaces, basic linings in  $\frac{1}{4}$ -ton furnaces rarely lasted more than a few casts, after which they cracked and metal frequently struck the coil. Some linings did not even survive the first cast, lack of sintering resulting in the lining material falling in as soon as the steel liner slid down into the melt formed.

*Grading:* So much work has been done on the grading both of monolithic linings and brick batches during the last 20 years that it is now hard to realise that even in 1928 the fact that sintering was largely controlled by the really fine fraction was not commonly known. As the result of much study based initially on Hugill and Rees' work on silica bricks, a standard (45 per cent. coarse—10 per cent. medium—45 per cent. fines) grading was developed which has since been the starting

point not only for numerous new monolithic linings, but also for brick batches required to yield a dense product. As will be seen from the figures given in Table LI the 45 per cent. of fines is further broken down

TABLE LI  
GRADING (PER CENT.) OF BASIC INDUCTION FURNACE  
LININGS

B.S.I. sieves	Tyler sieve equivalents	Delivery No.					
		1	2	3	4	5	6
On 7 mesh	On 8 mesh	12.0	14.8	10.5	8.3	10.0	8.9
7-25 "	8-28 "	34.7	30.5	37.0	35.7	34.0	34.6
25-72 "	28-65 "	19.0	19.1	21.7	23.1	21.0	23.1
72-150 "	65-150,	12.7	12.5	11.9	12.0	14.0	12.9
Through 150,,	Through 150,,	21.6	23.1	18.9	20.9	21.0	20.5

into about 20 per cent. ordinary fines (72 to 150 mesh) and 25 per cent. super fines (through 150 mesh). If this latter fraction is insufficient trouble is likely to be experienced due to lack of sintering, but if on the other hand it is excessive, the lining is likely to be extremely difficult to ram, the ramming tool piercing it almost as though it were a liquid. Somewhat coarser linings have been successfully used, but it should be noted that the presence of too much "on 7 mesh" material may lead to serious segregation and, therefore, to localised porosity. The samples reported in Table LI were taken from consecutive deliveries and show that with reasonable care grading can be kept within fairly close limits. This particular material is rather coarser than standard and, therefore, requires special care to avoid segregation. For this reason all the material required for a lining should be premixed and the ramming process carefully watched so that any tendency to segregate is observed. That segregation does occur readily with much material can be simply demonstrated by bumping a box containing it on the bench, when it will be seen that the coarse fraction rises to the top or side of the box forming a pocket of highly porous material.

*Chemical analysis:* A great deal of research has been done on induction furnace linings, but the basic material found most satisfactory in Great Britain was nevertheless developed somewhat empirically. Although it consisted in the main of Austrian and Grecian dead-burned magnesites, it also contained sand and other additions, whose precise influence was not appreciated until serious trouble occurred during the



Second World War. Trials made with alternative materials, *e.g.*, sea-water magnesia, bonded with boric acid, had given entirely satisfactory results in 5 cwt. furnaces, but when such material was used in 2-ton furnaces a spate of breakouts occurred (*see* later section on firing shrinkage). These were at first thought to be due to the sea-water magnesia itself, but it was subsequently shown that the latter was quite satisfactory provided the right additions were made to it. Needless to say all magnesite used for this purpose should be well-dead-burned, the specific gravity test forming for each type of material a useful yardstick. Corrections must of course be made for such things as the iron oxide content, a specific gravity of 3.50 for low iron Greek magnesite being comparable with, say, 3.56 for Austrian magnesite containing 4 to 8 per cent. of ferric oxide. It might well be expected that still better results would be obtained by the use of fused magnesia, since it is both pure and highly crystalline. Trials carried out on a  $\frac{1}{2}$ -ton furnace, however, using a (45-10-45) type grading, and made both with and without boric acid bonds, did not show any better life than was obtained with normal magnesite linings, whilst shrinkage cracking appeared to be rather worse than usual.

*Sintering tendency:* The importance of the fine fraction in promoting sintering was first fully realised as a result of the complete failure of a Canadian magnesite lining installed in a British furnace. Subsequent examination showed that this particular lining only contained 5 per cent. of material through 120 mesh. As the furnace heated-up and the liner slid down into the bath, the unsintered powder at the top fell in leaving a bare coil. Laboratory tests carried out on this material, on satisfactory material used elsewhere, and on the faulty material reground to give more fines, suggested that the trouble was purely one of inadequate grinding. Part of the original batch was, therefore, reground and a second lining installed. This proved entirely satisfactory and was an important step in the development of the (45-10-20/25) type grading.

Most of the basic linings in use today contain low melting point bonds, such as boric acid, and in consequence show adequate sintering at quite low temperatures, *e.g.*, 1000°C. If there is any doubt as to whether a new material is likely to sinter adequately in practice, it should be compared with the previous standard, by filling small crucibles with the two materials and heating them side by side to increasing temperatures.

*Firing shrinkage:* It was generally thought until about 1940 that provided the high temperature shrinkage (1 hour at 1600°C.) on a basic lining could be kept below, say, 4 per cent., no trouble would be experienced due to cracking even with large induction furnaces.

Disillusion came with a rather sudden changeover from mixtures of Austrian and Greek magnesite to sea-water magnesia as the base material. Linings made from the former led to a spate of "strikes to the coil", although their high temperature shrinkage was markedly lower than normal. A valuable clue to the cause of the trouble was afforded by the casual remark of an operator that he had no confidence in a basic lining unless it showed serious cracking at the surface. Since avoidance of cracking was the object of the lining producer there could only be one explanation, *viz.*, that the bad cracks observed did not go all the way through the lining. It was, therefore, decided to carry out shrinkage tests over a range of temperatures and in addition to see whether the presence of sand and boric acid known to have been added to certain commercial batches had in fact any influence on firing shrinkage. The results given in Table LII, and shown diagrammatically in fig. 226, provided a dramatic answer. They

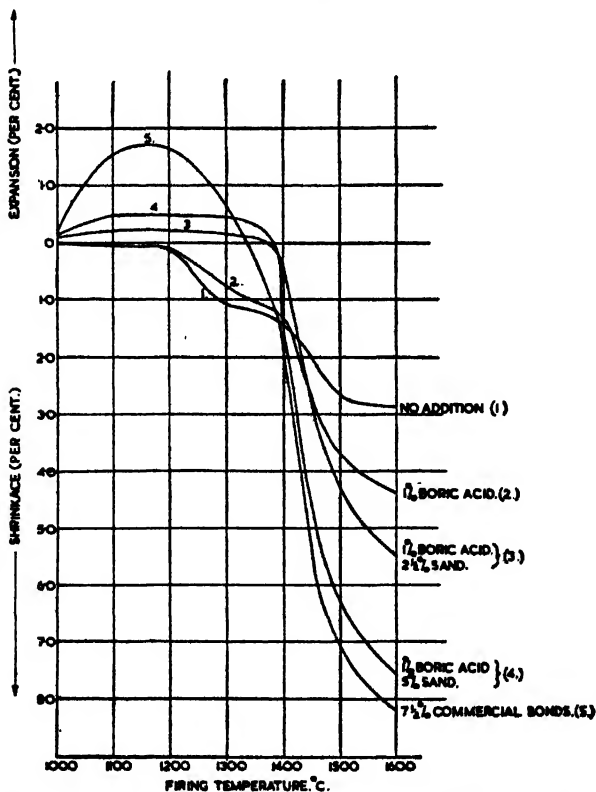


Fig. 226. Effect of boric acid and sand on the firing shrinkage of sea-water magnesia.

TABLE LII  
EFFECT OF SAND ON THE FIRING SHRINKAGE OF SEA-WATER MAGNESIA

<i>Sea-water magnesia plus:</i>	<i>Linear change (%) on firing 1 hr. at:</i>						
	1000°C.	1100°C.	1200°C.	1300°C.	1400°C.	1500°C.	1600°C.
No addition .. ..	0.0	-0.04	-0.09	-1.11	-1.38	-2.73	-2.90
Boric acid, 1% .. ..	0.0	-0.04	-0.08	-0.72	-1.30	-3.72	-4.40
Boric acid, 1%; sand, 2½% ..	+0.04	+0.20	+0.20	+0.16	-0.25	-4.37	-5.5
Boric acid, 1%; sand, 5% ..	+0.11	+0.47	+0.43	+0.47	-0.31	-6.30	-7.6
Commercial bonds, 7½% ..	+0.08	+1.51	+1.66	+0.61	-1.45	-7.59	-8.2

showed that whereas a straight magnesia lining, or one merely bonded with boric acid, gave a small but definite shrinkage even at 1200°C. successful commercial linings, or magnesite batches containing the known commercial bonds, showed a permanent expansion at this temperature of over 1½ per cent. The results obtained at 1600°C., however, showed far higher shrinkage with these additions than with straight magnesite or magnesite with boric acid alone. The main deductions drawn from this work, which have been fully reported by Chesters, Lee and Mackenzie, in their 1949 paper on "Steel-tight Linings for Basic Induction Furnaces", was that high temperature shrinkage is not serious *provided* such linings also show a positive expansion at an intermediate temperature, such an expansion leading to compression stresses in the intermediate zone of the lining and consequently to steel tightness. It remained to provide a reasonable explanation of the low temperature expansion and, therefore, a logical method of controlling it. X-ray examination and detailed microscopic work showed that when commercial linings containing borax and sand were heated up the sand first dissolved in the borax and the siliceous glass so formed then reacted with the magnesia to form a forsterite coating on the grains which forced the latter apart. A permanent expansion was thus obtained at the expense of increasing porosity. As the temperature was further raised the forsterite redissolved in the melt giving magnesia and a large quantity of flux which explains the substantial shrinkage at 1600°C. That even greater expansions can be obtained by increased or modified bond is shown by Table LIII, and by fig. 227, where it is shown that an expansion of over 6 per cent. at 1200°C. can be obtained by adding 10 per cent. borax and 20 per cent. sand but only at the expense of a very high and, therefore, undesirable shrinkage at 1600°C.

Once it had been established that low temperature expansion was desirable in order to minimise "strikes to the coil", a system of quality control was established (*see* fig. 228) and the suppliers warned whenever a point approached the lower  $2\sigma$  (1 in 40) limits. This system of expansion control has been in use ever since and has led to a virtual elimination of such failures.

*Slag resistance:* Although the lining itself must provide the final test, it is always wise to carry out preliminary checks on any materials of a radically new type. This can conveniently be done by one of the following methods:

(1) *Small induction furnace tests*

Crucibles made from the new lining material are tested either in the unfired or pre-fired condition. They are placed in

TABLE LIII  
EFFECT OF VARIOUS FLUXES ON THE FIRING SHRINKAGE OF SEA-WATER MAGNESIA

Sea-water magnesia plus:	Linear change (%) on firing 1 hr. at:								
	800°C.	900°C.	1000°C.	1100°C.	1200°C.	1300°C.	1400°C.	1500°C.	1600°C.
No addition ..	0.00	0.00	0.00	-0.13	-0.40	-0.41	-1.53	-2.06	-2.96
Boric acid, 1% ..	+0.10	0.00	-0.05	0.00	-0.14	-0.29	-1.22	-2.13	-3.48
Borax, 2%; sand, 4% ..	0.00	0.00	+0.19	+1.78	+2.33	+2.47	+0.37	-2.60	-5.92
Borax, 1%; boric acid, 1%; sand, 4% ..	+0.10	0.00	+0.42	+2.35	+3.08	+3.29	+1.67	-1.76	-4.57
KF, 2%; sand, 4% ..	+0.53	+0.60	+0.69	+0.91	+1.02	+1.00	+0.46	-0.73	-4.34
CaF <sub>2</sub> , 2%; sand, 4%; ..	+0.25	+0.25	+0.86	+2.82	+3.67	+3.87	+3.15	-0.27	-3.94
Cryolite, 1%; sand, 4% ..	+0.22	+0.27	+0.46	+1.09	+1.76	+1.88	+0.97	-2.00	-5.46
Cryolite, 2%; sand, 4% ..	+0.43	+0.46	+0.77	+1.24	+1.47	+1.45	+0.56	-2.87	-5.38
Na <sub>2</sub> CO <sub>3</sub> , 2%; sand, 4% ..	+1.34	+1.46	+1.71	+1.71	+1.71	+1.71	+0.18	-2.33	-4.66
Borax, 2%; quartz (7-25 mesh), 2%; sand, 1%; silica flour (through 72 mesh), 1% ..	+0.21	+0.14	+0.54	+1.36	+1.82	+1.81	-0.57	-3.32	-5.96
Borax, 2%; quartzite (7-25 mesh), 4% ..	+0.31	+0.22	+0.45	+1.27	+1.81	+2.03	+1.02	-1.36	-4.77
Commercial sand, 7½% ..	+0.15	+0.13	+0.64	+1.34	+1.60	+1.29	-3.10	-5.30	-7.80
Borax, 10%; sand, 25% ..	+1.69	+2.00	+3.69	+5.78	+6.60	+4.76	-1.03	-5.54	-12.20
Commercial lining (Austrian-Grecian magnesite mixture) ..	+0.10	+0.13	+0.08	+0.80	+1.14	+1.14	-0.91	-2.81	-5.06

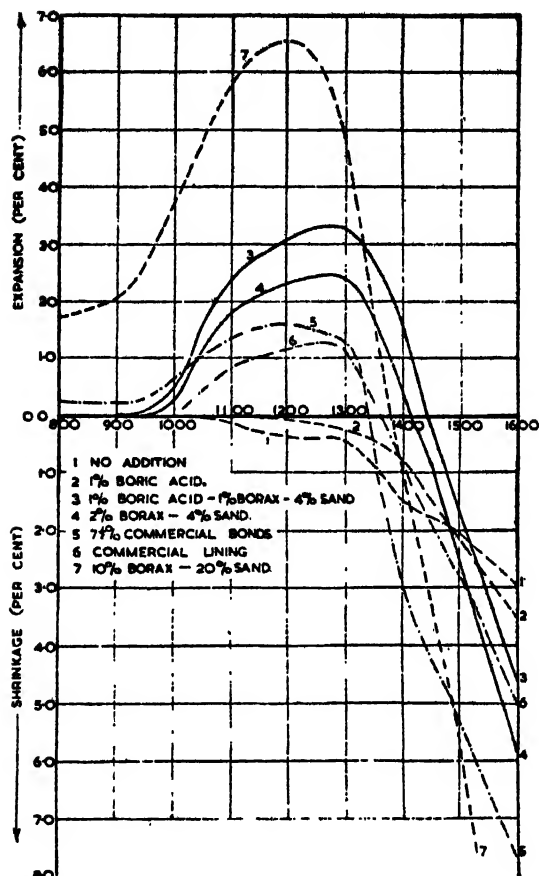


Fig. 227. Effect of boric acid, borax, sand and commercial bonds on the firing shrinkage of sea-water magnesia.

a small induction furnace and are charged with about 1 lb. of bar iron, to which can be added millscale or other fluxes if desired. After a given melting time the furnace is shut off, the cast poured, and the crucible examined. A comparison of the slag cut with that obtained on the standard material gives a first estimate of slag resistance. It can also be used to determine the probable effect on a standard lining material of a new slag.

## (2) Crucible-slag tests

Similar tests can be made in which the moulded crucibles are filled with a suitable slag, *e.g.*, iron oxide, and heated up in a gas-fired kiln. This method is not as satisfactory as an actual

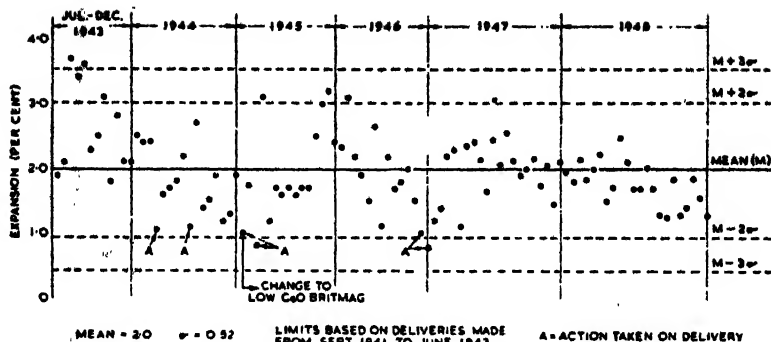


Fig. 228. Quality control chart for firing expansion (1-hour at  $1150^{\circ}\text{C}$ ) of a basic induction furnace lining.

induction furnace test, since slag composition tends to alter in the absence of metal and there is no temperature gradient through the crucible.

(3) *Melting point of refractory-slag mixtures*

The technique in which the melting point of a range of lining-slag mixtures is determined either in the form of small cones or pills has already been described in Chapter I.

*Other basic refractories*

An alternative method widely employed in the United States of obtaining volume stability in a basic lining, is the use of a magnesia-fused alumina mixture. As Chesters and Parmelee showed in 1934 the reaction of magnesia with alumina to form a spinel results in a very substantial expansion, which again is associated with an increase in porosity (see fig. 229 page 528). Actual data for a series of testpieces made from magnesia-alumina mixtures, ranging from 50/50 to 0/100 are shown in Table LIV and also in fig. 230. Although the maximum expansion was obtained with 70 per cent. alumina—30 per cent. magnesia mixtures, the reverse ratio, i.e., 30/70 gives adequate expansion and is more commonly employed.

Numerous other basic refractories have been tried, e.g., chrome-magnesite linings, both in brick and monolithic form. The results were quite good, but no advantage was obtained over magnesite or magnesite-alumina, and since with chrome there is always the risk of chromium contamination such linings were not pursued. Trials of specially graded doloma, using 1 per cent. boric acid as a bond, also gave satisfactory results, but the life obtained was appreciably less than with magnesia. From time to time trials have also been made of magnesia-zircon

TABLE LIV

INFLUENCE OF MAGNESIA/ALUMINA RATIO ON THE  
FIRING EXPANSION OF SPINEL-FORMING MIXTURES

<i>Magnesia Alumina</i> Ratio	Sieve sections (B.S.I.)					Linear firing expansion at 1600°C. 1 hr.      2 hrs.	
	7-25 m.	25-72 m.		Through 72 m.			
	$Al_2O_3$	$MgO$	$Al_2O_3$	$MgO$	$Al_2O_3$		
	%	%	%	%	%	%	%
50/50	45	5	5	45	—	2.7	6.8
40/60	45	—	10	40	5	6.3	8.3
30/70	45	—	10	30	15	7.4	9.4
20/80	45	—	10	20	25	3.3	3.3
10/90	45	—	10	10	35	0.7 (Sh)	0.3 (Sh)
0/100	45	—	10	—	45	1.9 (Sh)	2.2 (Sh)

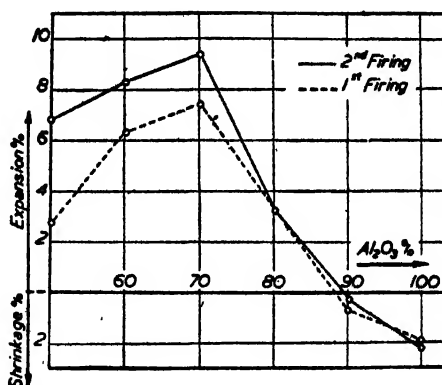


Fig. 230. Linear expansion or contraction of magnesia-alumina mixtures after one and two hours at 1600°C.

linings, which also show a positive expansion on firing. Here again, however, the slag resistance is less and the applications, therefore, limited.

### Neutral refractories

Straight chrome bricks were tried in the unfired form during some of the earlier induction furnace trials, with very striking results. After only one heat the chrome bricks, which were set spirally as a series of panels in a magnesite lining, had largely dissolved in the bath, leaving a series of steps up the lining face. Subsequent analysis of the steel showed that virtually the whole of the missing chromium had gone into the bath.



Numerous workers have also employed linings of zircon (zirconium silicate) though with very varied results. The primary difficulty here would appear to be one of providing a satisfactory bond, the use of materials such as fireclay resulting in rapid attack on the matrix and the floating out of undissolved zircon into the slag. Even if a bond could be found to keep the zircon in position it is doubtful whether its resistance to iron oxide attack would be anything like as great as that of magnesia.

### *Acid refractories*

By far the most commonly used acid lining is ganister or quartzite bonded with boric acid or borax. Substantial use has also been made on the Continent of natural clay bonded sands, and more recently of a mixture of pure quartzite with approximately one-third the weight of an impure quartzite whose flux content is just sufficient to provide the necessary sintering. Where ganister or quartzite is employed, it may be used raw or calcined, whilst the amount of boric acid or borax added is generally of the order of 1 per cent.

*Grading:* Typical gradings of acid lining materials are given in Table LV and in fig. 231. The particular samples shown were selected to indicate the very serious risk of segregation that may occur in packing a dry granular material in bags, particularly when an

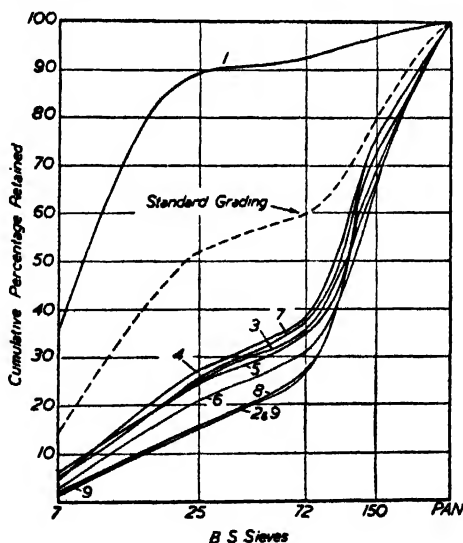


Fig. 231. Gradings (1 to 9) of acid induction furnace lining (one delivery) showing abnormally bad segregation during bagging.

TABLE LV  
 VARIATION IN GRADING (PER CENT.) OF COMMERCIAL DELIVERIES OF ACID INDUCTION  
 FURNACE LINING

Sample No.	1	2	3	4	5	6	7	8	9	10*
B.S.I. sieves	0	0	0	0	0	0	0	0	0	0
On 1"	36.0	2.0	5.0	4.5	5.5	2.5	5.5	2.5	1.5	14.0
1"-7 mesh	53.5	13.0	20.0	22.5	19.0	18.0	20.0	13.0	13.5	38.0
7-25 "	3.0	11.0	10.5	11.0	10.0	10.0	11.5	11.0	11.5	8.0
25-72 "	4.5	47.0	34.0	37.5	38.0	37.0	39.0	41.5	46.0	18.0
72-150 "	3.0	27.0	30.5	24.5	27.5	32.5	24.0	32.0	27.5	22.0
Through 150 "	..	..	..	..	..	..	..	..	..	..

\* Standard grading.

appreciable amount of coarse ("on 7 mesh") fraction is present. Since it has been shown that acid linings containing as much as 1 per cent. of water can be used without any risk of breakdown in coil insulation and since such a moisture content reduces both segregation and the risk of silicosis, the addition of a small quantity of water is strongly recommended.

*Chemical composition:* The satisfactory results obtained with Klebsand linings are presumably due in large part to their physical properties, since, as the analyses given below show, the Klebsand contains over 8 per cent. of alumina, which must have a very substantial effect both on its melting point and its resistance to slags high in iron oxide:

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Borax	Loss on Ignition
Ganister lining ..	96.8%	0.5%	0.4%	0.2%	1.4%	0.5%
Klebsand lining ..	87.4	8.6	0.2	0.3	nil	2.9

*Sintering tendency:* Tests made by the crucible method already referred to under basic linings, show that acid linings containing a normal amount of fines and bonded with boric acid sinter readily at 1000°C.

*Firing shrinkage:* The shrinkage problems experienced with basic linings are quite unknown with acid linings, no trouble being experienced with "strikes to the coil" even with 5-ton furnaces. Indeed the first silica linings were usually made with crushed silica bricks in order to avoid possible damage to the coil by excessive firing expansion. Tests made on acid lining materials have shown expansions as great as 10 per cent. on a testpiece fired at 1600°C., but the expansion in practice is likely to be far less both because flux penetration will tend to reduce expansion at the working face and because laboratory data is liable to be misleading due to the increased expansion caused by cracking of the testpiece.

*Slag resistance:* The resistance of silica to slags high in iron oxide is markedly lower than that of magnesia, and it is mainly the desirable physical characteristics of acid linings that have resulted in their continued use. High manganese steels and fluorspar slags must be avoided, as the risk of sudden failure due to slagging or melting is much greater than with magnesia. On one occasion, when the charge in an acid lining "bridged", most of the bottom melted and rose to the top as a large pool of silica glass.

### (c) LIFE AND CAUSES OF FAILURE

No particular difficulty now exists in producing basic linings that will do a full week's work on alloy steel production. For a  $\frac{1}{2}$ -ton furnace this means, say, 85 casts, and for a 2-ton furnace nearer 55 casts.

These figures correspond roughly for both sizes of furnace to a lining consumption of 12 lb. per ton. Certain of the materials available are satisfactory for longer periods than this, but unless double this life can be guaranteed, no real benefit is obtained, since such a life would inevitably mean a mid-week shut-down. The actual life obtained varies greatly with the steels being made and even the succession in which they are produced. Thus it is stated that a series of low carbon heats, followed by a series of high manganese heats, is much less damaging to the lining than when the two qualities are made alternately.

Acid linings have a similar life to basic on ordinary qualities and can be used with far less risk on larger size furnaces. Furthermore the life obtained can be considerably extended by periodic patching with a mixture of ganister and fireclay. Incidentally basic linings are also sometimes patched by mixing lining material with water glass.

Although a strike to the coil is still the biggest risk with an induction furnace, such failures now occur comparatively rarely and a warning of such a break through can be given by means of an electric "tell-tale" consisting essentially of an earth leakage relay. So sensitive is this instrument, which can be made to work a cut-out when the leakage exceeds a safe figure, that it has to be desensitised during the burning in of a damp lining.

Most linings wear out due to a combination of erosion and corrosion, which are usually worst at the slag line. The rate of wear would doubtless be far greater were it not for the extremely steep temperature gradient existing through the lining and which incidentally is beautifully illustrated by a section cut through an acid lining. Such a section shows the zoning, so characteristic of open-hearth furnace roofs, *viz.*, a working face of cristobalite, a comparatively unaltered back face and an intermediate zone high in tridymite. Periodically abnormal failures occur due to other causes, *e.g.*, inadequate bonding or insufficient fines. They may also occur due to local segregation of coarse material with a consequent high permeability. Tramp metal or other contamination may also be a cause of trouble, but such inclusions will normally be noticed when the material required for a lining is remixed on the stage prior to ramming. With acid linings particular care must also be taken to avoid prolonged "bridging", *i.e.*, welding together of the charge, since otherwise a liquid bath formed below may shortly become superheated to the point at which it can melt a substantial part of the lining material.

#### (d) LINES OF IMPROVEMENT

The main problem still requiring solution is that of the basic lining suitable for use in really large induction furnaces. Given such a lining

greater security would also be obtained in the smaller sizes. It is possible than an answer may come to this problem through the use of spinel type linings containing a higher alumina content than is at present employed.

Other problems still remaining, though not serious, are the reduction of segregation during ramming and improvement of the joint between the top of the liner and the furnace spout.

#### BRICK LININGS FOR CORELESS INDUCTION FURNACES

##### *(Method 4)*

As early as 1931 trials were made both of unfired and fired bricks in small induction furnaces. The results obtained with this method were quite satisfactory but the care needed to ensure perfect joints made general use distinctly dangerous. More recently trials have been made with machine pressed curved tiles of far greater strength, and it is possible that this may well prove their value in large furnaces, where an adequate thickness of monolithic material can be used between the tile and the coil. That such trials can be extremely useful in development work was shown very early on, when successive rings were built of different quality bricks and the relative wear resistance compared. One such lining made with Grecian magnesite merely bonded with gum to give green strength proved exceptionally good, having a life of 115 casts at a time when even 100 was remarkable. Considerable work has also been done with acid linings made from unfired silica brick batch of normal steelworks grading.

The real difficulty with this type of lining, however, still lies in ensuring a sound installation. All the bricks or tiles used must be free from obvious defects, and great care must be taken to see that the first course is concentric with the coil and its top face truly horizontal, since otherwise the cylindrical lining will tend to lean in one direction or another and any attempt to correct such deviation is bound to lead to open joints. There is little doubt that given a fairly thick lining, say 3 in. minimum, the working face of which was built with heavily pressed tiles and the back rammed with normal first-quality lining material, a slightly longer life could be obtained than with the fully rammed job. Unless, however, the life of such a lining was several times that of the standard material there would be little incentive to use it in view of the added care required in installation and the additional risks associated with the joints.



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### Chapter XIII

## SOAKING PITS, REHEATING FURNACES AND HEAT TREATMENT FURNACES

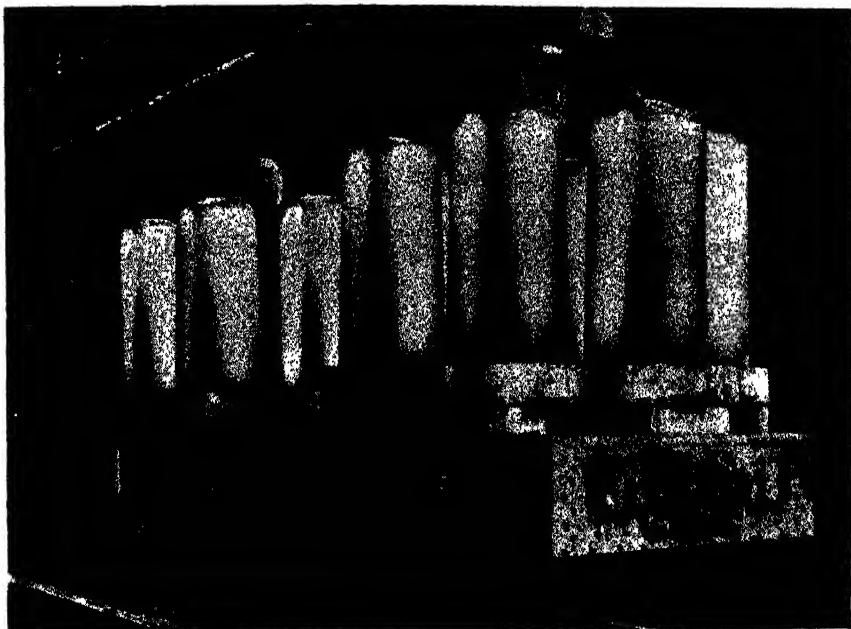
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**R**ELATIVELY little research has been carried out on refractories for soaking pit and reheating furnaces, presumably because the operating conditions are much less severe than in steelmaking furnaces, and in consequence there is no great difficulty in finding refractories that last long enough to avoid the risk of serious interference with production. The less severe conditions also mean relatively low refractories cost per ton of steel treated and a wider choice of materials capable of giving good service. There are, of course, exceptions to the above generalisation, for example, the hearths of large pusher type furnaces, such as are used for reheating large slabs, often prove difficult to maintain due to abrasion of the hearth by a moving charge, and the simultaneous attack of molten iron oxide, whilst certain of the more intense combustion type reheaters even demand the use of super refractories in the burner blocks because of the exceptionally high operating temperatures. It should not, however, be deduced from the above that research on soaking pit and reheating furnace refractories in general is not worth while, since substantial savings are still possible and the development of new materials, *e.g.*, tougher high temperature insulating bricks, might well react both on design and fuel consumption.

All the above furnaces can be considered as process units, that are employed either to prepare steel for subsequent rolling, forging, or shaping, or to heat treat it in order to obtain desirable metallurgical properties (*see* figs. 232, 233, 234 and 235, pp. 549 and 550). From the design angle they can be conveniently sub-divided into two main groups, *viz.*:

1. Batch furnaces, in which the temperature is nominally constant throughout the furnace but varies with time.
2. Continuous furnaces, in which the temperature varies along the furnace but is nominally constant at any point.

From the standpoint of refractory selection, however, it is better to classify such furnaces according to their function. In the present discussion, therefore, the following grouping will be employed:

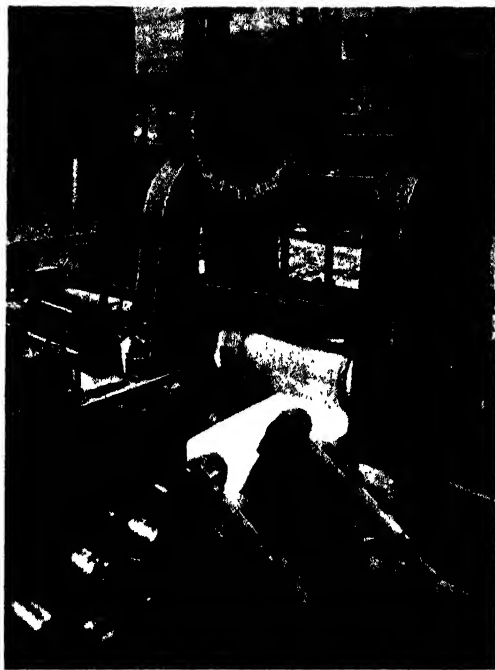


*Fig. 232. Two plates of bottom poured ingots on their way to the soaking pits.*

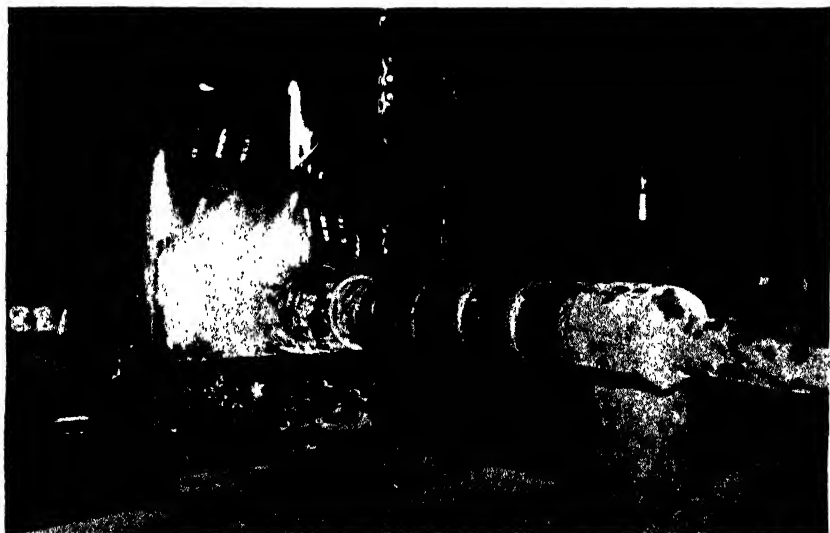


*Fig. 233. Billet mill rolling ingots from the soaking pits.*





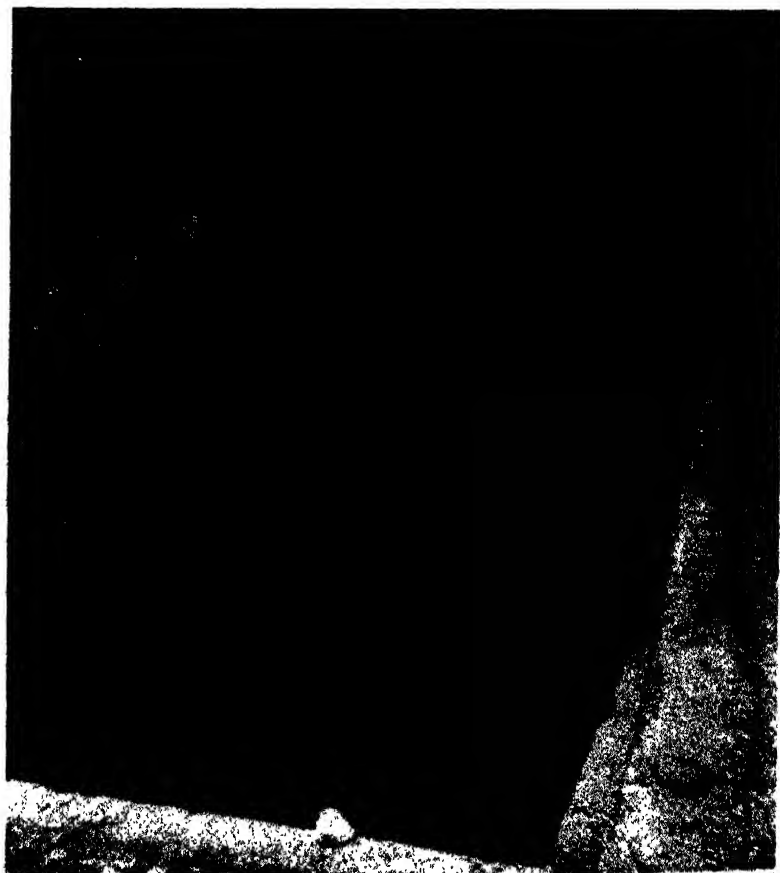
*Fig. 234. Electrically-driven slab mill rolling ingots after treatment in the soaking pits.*



*Fig. 235. Withdrawing a collared forging from a reheating furnace.*



*Fig. 236. Soaking pit installation showing roof removal mechanism (courtesy Stein and Atkinson Ltd.).*



*Fig. 237. Stein and Atkinson soaking pit after 2 years' operation.*

(a) *Soaking pits*

Used for heating of ingots and large forgings. Operating temperature range 1200° to 1350°C.

(b) *Reheating furnaces*

(i) Continuous:

Used for heating of small ingots for rolling and forging but more usually for reheating semi-finished products for further deformation, *e.g.*, billets and slabs into bar, rod and strip, also smaller forgings.

Operating temperature range 1050° to 1250°C.

(ii) Batch:

Used mainly for reheating prior to forging and shaping.

(c) *Heat treatment furnaces*

A multiplicity of designs for various purposes, including batch, continuous pusher, roller hearth and walking beam types.

Operating temperature less than 1000°C.

## SOAKING PITS

The soaking pit offers an extremely good example of the way in which the engineer can sometimes come to the rescue of the refractories' technologist. Even 20 years ago soaking pits in common use were very little more than "holes in the ground", which were battered or slagged to death in a matter of months. The modern soaking pit on the other hand both is and looks like an engineering device (*see* fig. 236, p. 551), and looks better after 2 years' operation (*see* fig. 237, opposite) than the old pits did after a couple of weeks. In these earlier pits the lids were merely a frame or clamp in which bricks were fitted and which frequently fell apart after repeated dropping by the crane. The ingots also were often dropped and impacted heavily on the side-walls, whilst the pit bottom was frequently inches deep in molten scale, which rapidly dissolved the adjacent brick and soon undermined the side wall. To complete the damage the slag was frequently removed by turning a hose into the pit at the week-end, thereby cracking up not only the solidified iron oxide but also the brickwork lining.

The modern soaking pit (figs. 238 and 239) is still essentially a cubic or cylindrical box, but is normally built with a ventilated steelwork foundation and is equipped with excellent gear for lifting and replacing the roof. The precise design and method of firing varies considerably, the features of the more common types being summarised in Table LVI.

The size and, therefore, the capacity of soaking pits varies widely, a typical range being 2 to 16 ingots, or 15 to 120 tons. Only about one-third

TABLE LVI  
SOAKING PITS

<i>Type</i>	<i>Classification</i>	<i>Design features</i>	<i>Notes</i>
I	Reversing regenerative (i) Mechanical reversal (ii) Isley controlled	Air regeneration and fuel gas recuperation. Reversing system necessitates firing and exhausting at alternative ends	At one time the only type in use, but now superseded by recuperative pits except for blast furnace gas firing where high air preheat may outweigh other disadvantages
II	Top-one-way fired	Air or air + gas recuperation according to fuel. Burner and exhaust port in same end-wall. Horseshoe flame combustion space above ingots	Most popular design for small and medium ingots. Economical in space. Made up to 27' 0" x 8' 0" hearth area
III	Top-two-way fired	Similar to above but burner and exhaust ports in both end walls off-set	Used as alternative to design IV up to 18 ft. square for large slab ingots
IV	Bottom-fired pit	Burner in hearth with exhaust at hearth level. Combustion space between ingots which are located around the central burner	Mainly used for slab and large blooming ingots
V	Bottom two-way fired pit	Similar to design III but burners about half-way down pit and combustion space between ingots	Rarely used
VI	Circular pit (tangentially fired)	Multiple burners firing round circumference with central exhaust port	High turbulence and large combustion space are claimed to give fast and uniform heating. The space requirements of this design are high
VII	Multiple fired rectangular pits	Numerous burners located in side walls between ingots	Not widely used

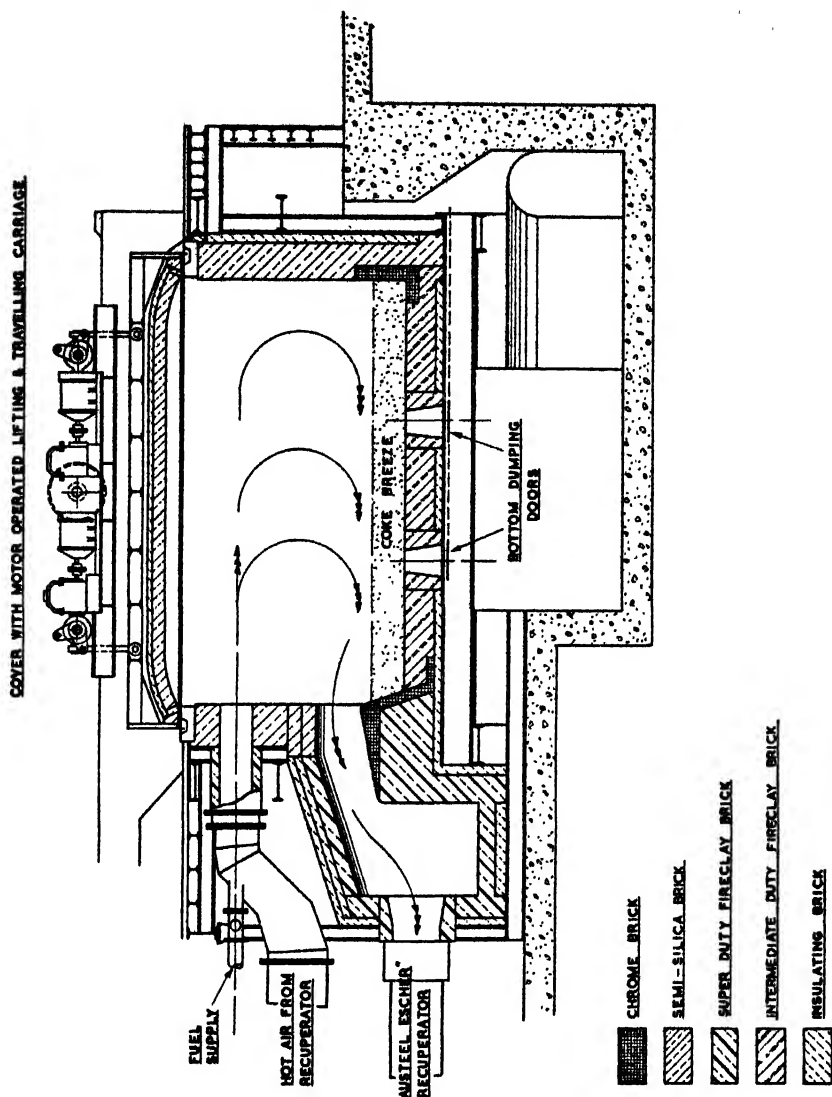


Fig. 238. Brick arrangement in soaking pit (courtesy Stein and Atkinson).

of the area of the bottom is actually occupied by ingots—too close a packing resulting in inadequate flow. The depth of the pit varies from about 8 ft. to 16 ft.

#### (a) ROOFS

These are generally of the suspended or 2-way end type construction, single lids tending to be preferred to rolling covers. The lids are all

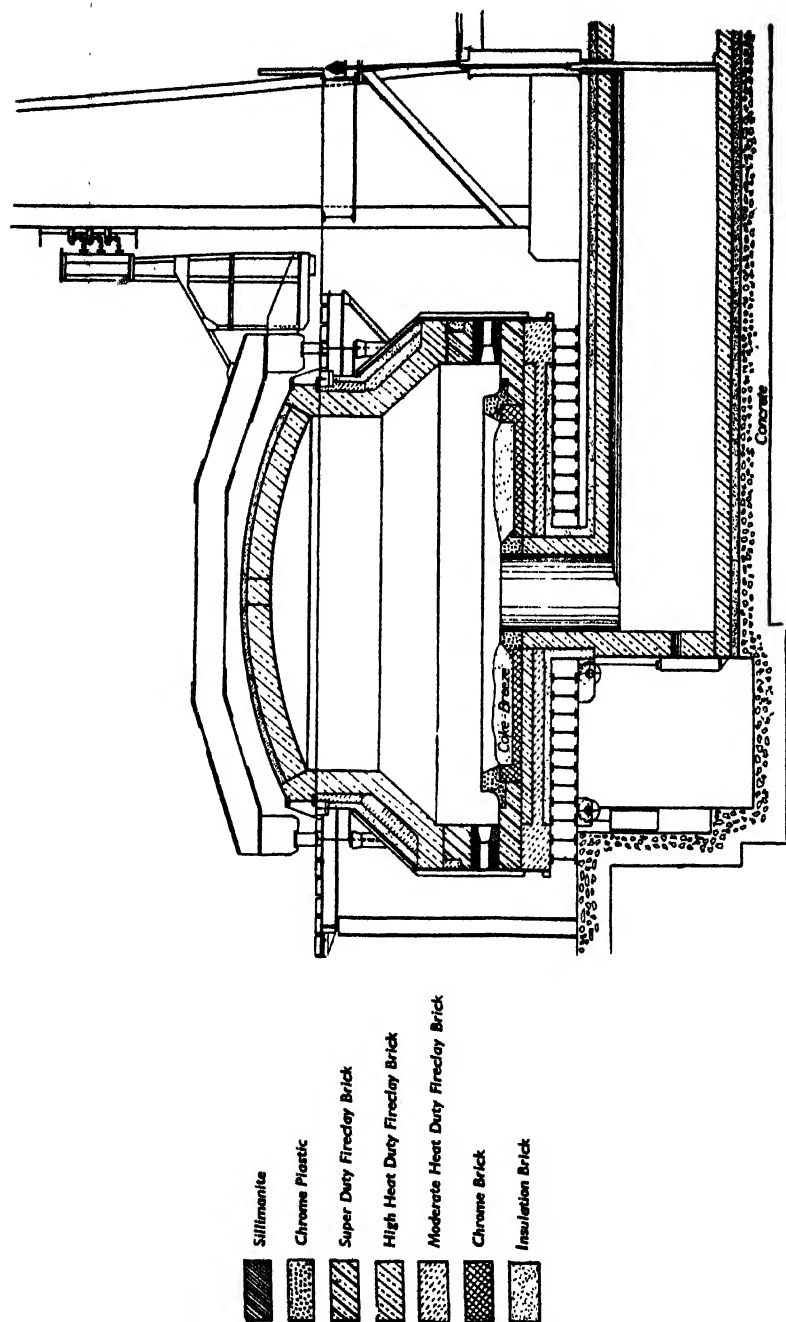


Fig. 239. Section through circular soaking pit showing materials used (courtesy Salem Engineering Co. Ltd.).

TABLE LVII

PROPERTIES OF SOAKING PIT AND REHEATING FURNACE  
ROOF BRICKS

	<i>Normal firebrick medium alumina fireclay</i>	<i>Special firebrick (suspended roof) aluminous fireclay</i>	
Code No. . . . .	F.2	F.40	F.8
Apparent porosity, per cent. . . . .	21.7	26.7	31.5
Bulk density:			
g.p.ml. . . . .	1.98	1.96	1.82
lb./cu. ft. . . . .	124	122	114
Apparent solid density, g.p.ml. . . . .	2.53	2.67	2.65
Crushing strength (on end)			
lb. per sq. in. . . . .	4140	790	n.d.
Permeability to air, c.g.s. units (perp. 9 in. $\times$ 3 in. face—1 skin) . . . . .	0.013	0.46	0.03
Permanent linear change on reheating, 2 hours at 1410°C. . . . .	—0.6 (contraction)	0.0	—1.3 (contraction)
Refractoriness, °C. . . . .	1680	1740	n.d.
Refractoriness-under- load. Rising tem- perature 50 lb. per sq. in.:			
Initial softening, °C. . . . .	1220	1220	1270
Rapid softening . . . . .	1450°	1290°	1400°
Fail temperature . . . . .	1540°	1530°	1480°
Thermal shock resistance . . . . .	30+	30+	30+
Thermal expansion (20° to 1000°C.) per cent. . . . .	—	0.55	0.41

arranged to have a vertical motion, in order to clear the sand-seals fitted around the periphery. From the refractory standpoint these latter are particularly important, since the good fit made possible by the use of a satisfactory sand-seal greatly reduces both the wear on the lids themselves and the damage to bricks at the top of the side-walls. The use of a tight fitting lid is also made from the standpoint of combustion,



since it enables a reasonable furnace pressure to be maintained and, therefore, minimises air infiltration. In many modern pits an automatic fuel cut-off operates whenever the lid is lifted, whilst replacement of the lid results in the fuel supply being automatically turned on.

Two main procedures are adopted for the lining of lids, *viz.*: bricking and the use of monolithic construction. Where bricks are employed they are normally of medium quality, such as, for example, the brick shown as F2 in Table LVII. At earlier installations bricks of far lower quality, *e.g.*, F17 in Table LVIII, were employed, but more recently there has been a trend to the use of either super-duty or high duty fire-clay bricks. Other operators, however, prefer a special silica brick, having a high thermal shock resistance, or a semi-silica (sand-clay) brick. Neither of these bricks possesses a thermal shock resistance anywhere near as good as that of fireclay, but their other properties, notably complete freedom from shrinkage, more than compensate. Trials have been made in soaking pit lids of high temperature insulating bricks, but the use of such materials although obviously desirable on grounds of fuel economy is still very limited.

The refractory used in monolithic construction is generally of the grogged fireclay type, being one of a number of proprietary brands having reasonable strength both on drying and after firing. The tendency of such monolithic linings to flake has been largely overcome by the use of corrugated refractory hangers embedded in the ramming mass. Roofs of this type have given very satisfactory life, for example, of the order of two years. This type of construction is now increasingly general in the United States, but in Great Britain the brick lined lid is more common and spalling difficulties are in part minimised by the use of suspended block construction.

#### (b) HEARTHS

The old type hearth, in which the ingots stood in a pool of molten slag, is increasingly rare. The modern hearth has a bottom consisting of coke breeze, millscale or crushed doloma, whilst the amount of scale formed in the pit is greatly reduced by careful control of the furnace atmosphere. The bottom below this layer is normally made up of firebrick with one or more openings for cleaning purposes through which the breeze or cinder can be dumped into cars placed under the pits. In one particular design the pit consists of a cast-iron "waffle" type lid, the cells of which are filled with insulating concrete. Used in conjunction with coke breeze covering it is stated that such bottoms may last as long as five years. Where a brick bottom is employed it is normally fireclay, but the quality may vary from super-duty through

TABLE LVIII  
PROPERTIES OF FIRECLAY BRICKS USED IN SOAKING PIT AND REHEATING FURNACE SIDE WALLS

Code No.	F.3	F.4	F.14	F.15	F.17	F.18	F.27	F.28
Apparent porosity, per cent.	20.8	20.8	26.9	34.5	23.7	23.7	18.9	22.0
Bulk density:								
g./p.ml.	2.09	2.09	1.93	1.79	2.00	2.00	2.00	1.99
lb./cu. ft.	131	131	121	112	125	125	128	125
Apparent solid density, g./p.ml.	2.65	2.65	2.64	2.74	2.63	2.63	2.53	2.56
Crushing strength, on end								
lb./sq. in.	1690	4510	3130	1740	6910	3320	1890	2940
Permeability to air—c.g.s. units (perpendicular grain. $\times 3$ in. face—1 skin)	0.015	0.027	0.0033	0.0097	0.0036	0.0098	0.40	0.029
Permanent linear change on reheating, 2 hrs. at 1410°C.								
..	+4.0 (Exp.)	+8.7 (Exp.)	+5.1 (Exp.)	-3.0 (Con.)	+3.6 (Exp.)	+3.5 (Exp.)	+1.1 (Exp.)	+0.06 (Exp.)
Refractoriness, °C.	1600	1620	1560	1630	1550	1560	1760	1710
Refractoriness-under-load. Rising temperature, 50 lb. per sq. in.:								
Initial softening, °C.	1240	1220	1100	1270	1010	1160	1270	1180
Rapid softening ..	1390	1350	1300	1340	1420	1380	1410	1370
Fail temperature ..	1520	1500	1450	1460	1520	1490	1600	1540
Thermal shock resistance ..	30+	26+	14	9	12	12	30+	30+

high-duty to a more modest quality, such as that shown in Table LVIII as F3. Basic bricks are also employed either with or without a covering of plastic chrome.

The coke breeze, which could itself be considered as a refractory, may be present to a depth of as much as 18 in. Its properties like those of the refractories which support it are considered important, *e.g.*, it is suggested that it should consist essentially of  $\frac{1}{4}$  in. to  $\frac{1}{2}$  in. material and that the ash fusion temperature should exceed 1200°C. In modern pits the breeze is dumped out at regular intervals, say every 14 days, the time taken being only a matter of half-an-hour. With cinder bottoms a layer of 2 in. to 3 in. of breeze or sand is spread on the brickwork of the pit bottom and the scale allowed to accumulate to a depth of over 1 ft. After several months' operation the pit is cooled and the cinder removed.

#### (c) SIDE WALLS

The precise design of this part of the soaking pit varies greatly with the particular type of installation, which in turn determines, for example, the type and location of both the burner blocks and exit flues. High structural stability, which is normally achieved by the use of thick walls, is essential, particularly since ingots frequently lean on the walls and may be dragged against them during subsequent lifting. Many American pits have been lined with a siliceous firestone (*see* Table V, Chapter II) as used in the lining of acid bessemer converters. This type of construction has one great advantage, *viz.*: that the large blocks employed (*e.g.*, 8 ft.  $\times$  2 ft.  $\times$  3 ft.), are far less liable to be damaged by abrading ingots than brick courses built from standard squares. In view of the success obtained with this material, it is not surprising that special silica brick of high thermal shock resistance, or semi-silica brick (sand-clay) are also found to give good service. Probably the most generally used material is, however, firebrick, which again varies in quality from the super-duty, through high duty, to more ordinary qualities. Thus the bricks given in Table LVIII as F3 and F27 have both given satisfactory service.

At certain positions in the side-walls, notably at the point where the ingots are liable to touch the wall, and at the base where liquid slag frequently tends to cause under-cutting, special refractories, usually of the basic type, are frequently employed. Thus at the slag line use has been made of chrome-magnesite bricks (both fired and unfired), chrome-silica bricks, forsterite bricks and high alumina bricks. The properties of such products are given in the appropriate materials chapters, or in Appendices 14 and 16.

*Recuperators and regenerators for use with soaking pits*

Where regenerators are employed, the refractories used are not dissimilar to those employed in the open-hearth furnace. Since working conditions are, however, much lighter no particular difficulties are experienced.

With recuperators on the other hand the problem is a very different one, in that gas-tightness is essential and all the materials used must, therefore, be free from shrinkage in addition to possessing other desirable properties, notably high conductivity and good thermal shock resistance. Where recuperators are of the refractory type, good quality hollow fireclay or silicon carbide tiles, are normally employed, but in recent years there has been an increasing tendency to use metallic recuperators, *e.g.*, those of the Escher type. Although the properties of silicon carbide (*see* Appendix 16) are rather remarkable, in particular it has both a high conductivity and a high refractoriness-under-load, this is one of the few places in the steelplant where it is employed. As an indication of its potential strength it has been stated that bricks having a crushing strength of about 10,000 lb. per sq. in. at 1350°C. are available. In addition it has an extremely high thermal shock resistance.

## REHEATING FURNACES

The numerous types of reheating furnace can be classified according to whether they are of the pusher type, moving hearth continuous type or the batch type, whilst these main groups in turn can be broken down into sub-groups.

1. *Pusher type continuous furnace*

This group, in which the stock moves over a fixed hearth, can be sub-divided as follows:

## (a) Simple top-fired single zone:

Used for billets and slabs of about 3 in. to 4 in. thickness and having an output rate of about 30 tons per hour.

## (b) Top-fired 2-zone:

For billets and slabs up to about 3 in to 4 in. and having very high output rates.

## (c) Multi-zone:

In which both top and bottom firing are employed, usually in two zones and with a top-fired soaking zone. These furnaces are employed in wide strip and plate mills, where outputs of the order of 150 tons per hour are required. Units have been built with as many as 3 top and 2 bottom-fired zones (*see* fig. 240).

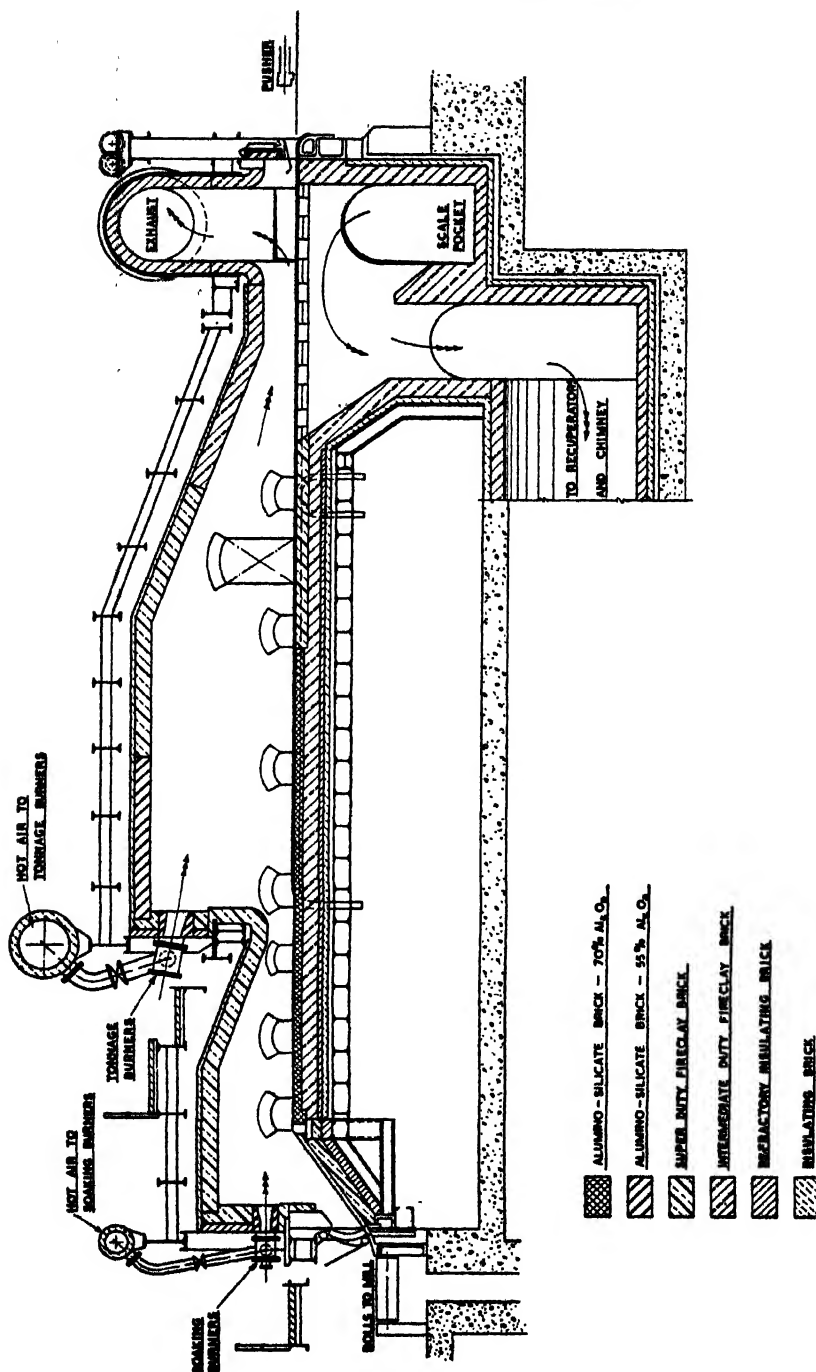


Fig. 240. Two-zone-fired continuous slab heating furnace (courtesy Stein and Atkinson Ltd.).

All the above furnaces may be side or end charged and side or end discharged. In the latter the stock slides down a sloping ramp, giving rise to substantial constructional and refractory problems. The hearths in such furnaces are usually constructed with raised skids, which may be either water-cooled or made from special materials, such as silicon-carbide, high alumina, or alloy steel. Where the furnaces are over 18 ft. in width, the roof is normally suspended. Side-walls are usually heavily insulated and frequently very much broken up by the presence of large numbers of relatively small burners.

## 2. *Moving hearth continuous furnace*

In this type the side-walls and roof of the furnace remain fixed, but the hearth moves straight forward or rotates. There are four principal sub-divisions, as follows:

### (a) Continuous bogie type:

Such furnaces take stock that could not be pushed directly, *e.g.*, tube billets or tyre and wheel ingots.

### (b) Rotary hearth type:

This is a variant on (a) above, but overcomes the heat loss from the bogies.

### (c) Walking beam type:

In which the hearth is made up of reciprocating elements normally covered with refractory tile, and the stock charged is light, *e.g.*, sheet, bar. Such furnaces are not recommended for use above 1100°C. because of the mechanical difficulties associated with high temperatures.

### (d) Roller hearth or continuous chain type:

This is a variant on (c) and, like the latter, is rarely used above 1100°C. (*see* fig. 241, p. 569).

## 3. *Batch type reheaters and forging furnaces*

These are usually simple box-type structures used for heating materials prior to forging or intermediate reheating between rollings, *e.g.*, in section and rail mills. They may or may not be equipped with recuperators and are charged and discharged either manually or by means of manipulated cranes or charging machines.

### (a) THE ROOF

The roofs of reheating furnaces vary greatly from simple sprung roofs of small furnaces to large suspended roofs of peculiar contour, such as are shown in figs. 240. Bricks suitable for use in the former type may

well not be suitable for use in the latter, where temperatures are higher and where the vital importance of maintaining production necessitates the use of the best available product regardless of initial cost. Again the question as to whether reheating furnace roofs should, or should not, be insulated can only be decided in relation to the particular installation concerned. Where difficulty is already experienced in maintaining a useful life it is generally best avoided, but where refractory life is excellent and insulation can be applied without risk of premature failure, its use is highly desirable.

Both the use of external insulation and the construction of complete roofs from high temperature insulation must, however, be justified on the grounds of fuel savings, which in turn necessitates the collection of satisfactory operating data. The reason for this is very simple, *viz.*: that insulation whether applied externally or in the form of hot face bricks almost always means increased initial costs and, furthermore, a decrease rather than an increase in furnace life. Other factors may, of course, enter into the calculation, in particular the greater speed with which the light-weight structure built of high temperature insulation can be brought into operation.

Where conditions are relatively mild, ordinary good quality firebrick, of the type shown as F.2 in Table LVII, give excellent results in roofs, whilst even lower qualities, *e.g.*, those shown in Table LVIII, as F.3, F.4, F.14, F.15, F.17 and F.18, may prove satisfactory. The ordinary good quality firebrick in Table LVII has many applications in the steelplant, apart from the reheating furnaces, being used in open-hearth furnace doors and in ladles. This brick has a fairly high bulk density (about 2.0), good crushing strength, a moderate permeability and reasonable volume stability at 1410°C. Its melting point is 1680°C., but it shows signs of softening under a load of 50 lb. per sq. in. even at 1220°C. This is one reason why insulation should always be applied with caution, since in such structures the load tends to be carried on the colder part of the brick, the thickness of which automatically decreases as insulation is applied. Another essential property of such a brick is good thermal shock resistance both in the "as received" condition and after heat treatment in service.

Where the operating conditions are more severe, and in particular where the span is greater and higher temperatures are employed, it may be necessary to go to super-duty firebrick, or their British equivalents, such as are given under the code number F.8 and F.40 in Table LVII. It will be seen that these bricks have a markedly higher refractoriness, though not under-load. When well fired they also show a very small permanent linear change on reheating. Under certain conditions

TABLE LIX  
PROPERTIES OF MISCELLANEOUS SOAKING PIT AND REHEATING FURNACE SIDE WALL AND DOOR BRICKS

Type	Code No.	Chrome-silica (soaker slag line brick)		Medium alumina door brick	Sillimanite burner and hearth brick
		X.1	X.2	F.5	X.14
Apparent porosity, per cent. . .	..	29.5	27.5	23.0	23.8
Bulk density: g.-p.ml. lb./cu. ft. . .	..	1.95 122	2.06 129	1.98 124	2.33 146
Apparent solid density, g.-p.ml.	..	2.77	2.84	2.58	3.06
Crushing strength (on end), lb./sq. in.	..	1360	1950	4110	3380
Permeability to air, c.g.s. units (per- pendicular 9 in. $\times$ 3 in. face—1 skin)	..	2.13	1.93	0.0086	0.025
Permanent linear change on reheating, 2 hrs. at 1410°C. . .	..	+0.1 (Exp.)	+0.1 (Exp.)	+4.8 (Exp.)	+0.05 (Exp.) (2 hrs. at 1500°C.)
Refractoriness, °C. (Seger cone)	..	1650 29	1570 20-26	over 1680 >30	— n.d.
Refractoriness-under-load (main- tained temperature), 25 lb. per sq. in., 1 hr. at 1400°C.) . .	..	0.3% collapse	Failed in 57 mins.	—	—
Rising temperature (50 lb. per sq. in.):	..	—	—	1140°C.	1500°C.
Initial softening . .	..	—	—	1260°C.	1580°C.
Rapid softening . .	..	—	—	1440°C.	1760°C.
Fail temperature . .	..	12	13	30+	30+
Thermal shock resistance	..	..	..	..	..



the use of bricks of far higher alumina content, *e.g.*, the sillimanite bricks shown as X.14 in Table LIX may be justified. It will be seen that the initial softening of this material takes place at a temperature several hundred degrees higher, that it is completely volume stable at 1500°C. and has a very high thermal shock resistance. Given relative freedom from corrosive atmospheres, *e.g.*, iron-oxide, it gives excellent service, and may under severe conditions justify its initial price, which is of the same order as that of basic bricks. For certain types of furnace, both in Great Britain and the U.S.A., use is made of special semi-silica (sand-clay) bricks, or special tough types of silica brick. Such materials have a lower thermal shock resistance at low temperatures, but given careful heating up yield an excellent reheating furnace roof, which rapidly takes on a glazed surface and remains extremely tight at the joints due to the after-expansion which such bricks normally possess. Furthermore, its refractoriness-under-load is such that it may safely be insulated, even though arches built of fireclay bricks of similar refractoriness would collapse if similarly treated.

Many reheating furnace roofs have a life of years rather than months and generally fail in the end due to spalling, which is frequently accelerated by shrinkage of the working face after prolonged high temperature treatment. The presence of dirt in the atmosphere, *e.g.*, ash from coal fires, may greatly accelerate such shrinkage and, therefore, also be an indirect cause of failure.

Future improvements in this field are more likely to come by the application of new bricks developed for other purposes, *e.g.*, cement kiln linings, rather than those developed specifically for reheating furnaces. The reason for this is simply that it is only where conditions are particularly severe that user demands lead to the carrying out of extensive research.

#### (b) THE HEARTH

Here again the conditions vary from those in which the temperature is low and there is little scale present, to those in which the temperature is particularly high and large quantities of molten scale drip from the charge on to the hearth. Under the less severe conditions a brick of the type shown as F.2 (Table LVII), and already described in connection with roofs, may be adequate, but the use of such a brick on any type of reheating furnace, where temperature is high and scale formation excessive, can lead to complete failure after only 1 or 2 heats. Thus one bogie-type furnace built with a firebrick hearth and loaded with ingots was found after the first heat to have the appearance of a ploughed field. This particular problem was largely solved by the use of a layer

of doloma on the hearth, but similar problems are more normally tackled by the use of either basic brick, chrome plastic or high alumina (70 per cent.) brick for the hearth surface. The amount of scale formed is in turn affected by the type of stock, the furnace atmosphere (oxidising or reducing) and the time-temperature schedule. It is normally of the order of 2 per cent. of the charged weight, but varies considerably both upwards and downwards according to operating conditions.

The other factor mitigating against long life, particularly in pusher type furnaces, is abrasion, which may be very substantial in furnaces taking heavy stock, *e.g.*, large slabs, particularly if the furnace is hard driven. Given a knowledge of the proposed operating conditions and, in particular, the temperature and the expected scale formation, a choice can be made either from the alumino-silicate range (38 to 70 per cent. alumina), or from the chrome or magnesite field. Under the worst conditions, *e.g.*, those in the large pusher-type furnaces, such as fig. 240, the final hearth is usually surfaced either with a chrome plastic or with 70 per cent. alumina type bricks. The life obtained with both materials varies greatly from plant to plant, but should normally be at least 6 months. Under similar conditions the difference between chrome plastic and 70 per cent. alumina would appear to be slight, some users find the one better and some the other. Magnesite chrome and chrome magnesite bricks, either fired or unfired, have also been used quite successfully, but where basic refractories are called for the use of plastic chrome would generally appear preferable. This material is of a proprietary type, about which relatively little has been stated in the literature. It can, however, be taken to consist essentially of chrome ore ground in a pan mill with relatively small amounts of clay and sodium silicate to give a plastic material suitable for ramming, and yielding a relatively tough product when fired at temperatures around 1350°C. Both the choice of the chrome, the grinding procedure, and the purity of the materials are, however, of first importance—even minor changes in composition making all the difference between a material which is tough at 1350°C. and one that is as weak as wet sand.

Chrome plastics are normally supplied in sealed tins and simply require to be rammed in position on the sub-hearth. Because they contain sodium silicate, which tends to form an impervious skin on drying, care must be taken to provide adequate venting of the hearth, since otherwise the steam formed during drying and heating-up may cause the working face to blow off. Such venting can be conveniently done by pricking the surface of the freshly rammed hearth at intervals with a pointed rod. Where slag attack is still a problem, but the abrasion conditions are less severe, other mixtures, *e.g.*, chrome-magnesite

gradings with ciment fondu as a bond, may be used. Such hearths can be dried out very rapidly and have been shown to give excellent results.

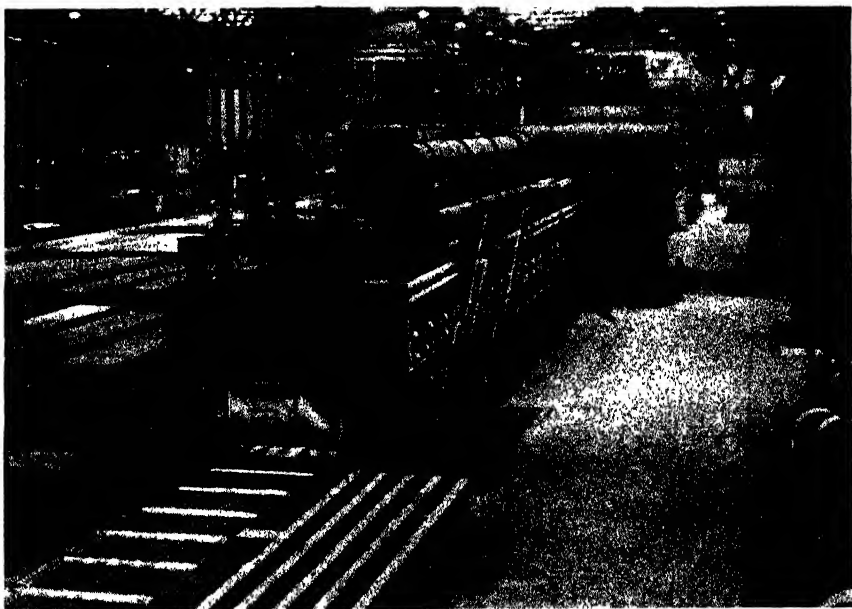
Where the abrasion conditions are such that even the best refractories have a limited life, recourse is normally made to skids, consisting either of alloy steel, or silicon carbide bricks set on edge. The success of these latter in positions where other refractories would undoubtedly fail, is presumably due in the main to their extremely high abrasion resistance and refractoriness-under-load.

In most reheating furnaces a measure of insulation below the hearth is not only desirable but quite safe, the only proviso being that the interface temperatures under equilibrium conditions do not exceed the safe limit for the particular insulating materials concerned. The choice of materials will not be dealt with here, as it has already been discussed at some length in Chapter VII.

#### (c) SIDE WALLS

The side wall problem in reheating furnaces is usually comparatively simple compared with that of the roof or hearth. The major complication lies in the fact that the wall is normally broken up by burner blocks and, in addition, is a composite one, consisting of a refractory face with one or more types of insulation on the outside. In the neighbourhood of burners this latter is dispensed with, partly because of constructional difficulties, and partly to reduce the strain on the burner itself. For many types of reheating furnaces, side walls can be quite satisfactorily built with fireclay bricks in the range 38 to 42 per cent. alumina, though, as with the roof, particularly severe conditions may necessitate the use of bricks of higher alumina content, *e.g.*, sillimanite. Basic refractories are rarely used in this position, though both special silica and semi-silica bricks are sometimes employed.

Even where the use of ordinary qualities is quite satisfactory in the side wall proper, special refractories may have to be employed for the burner blocks. Where the burners are of the low intensity types, super-duty quality, or sillimanite, may prove satisfactory, but with special high intensity burners, *e.g.*, the Selas type, super refractories may be required. In such positions even sillimanite has been proved inadequate and recourse had to bricks of 90 per cent. alumina and over. The doors in reheating furnaces present a similar problem to the rest of the side wall but with additional emphasis on high spalling resistance due to the frequent heating and cooling which occurs when the doors are opened and closed. The principal cause of deterioration in side walls is spalling, which, as in other positions, can be greatly aggravated by continued shrinkage of the refractories in service.



*Fig. 241. Roller hearth type normalising furnace used for heat treating plates (courtesy Gibbons Bros. Ltd.).*



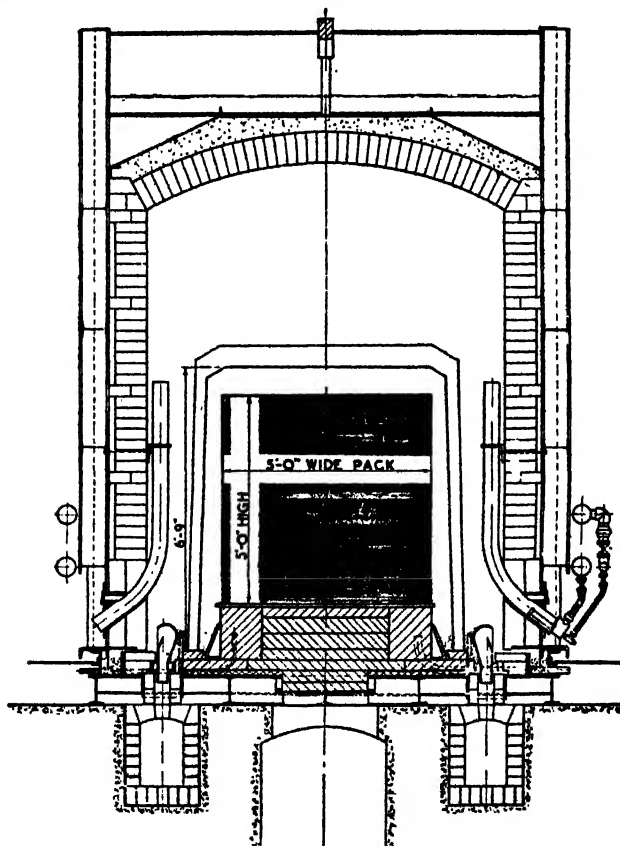
## RECUPERATORS AND REGENERATORS FOR USE WITH REHEATING FURNACES

These are similar in general type to those employed with soaking pits, though in general the operating conditions are less severe and, therefore, the refractories used in similar soaking pit installations quite satisfactory.

## HEAT TREATMENT FURNACES

There are hundreds of different types of heat-treatment furnaces, but these may be classified into three main types, *viz.*:

- (a) Continuous roller hearth furnaces for plate normalising (*see* fig. 241, p. 569).
- (b) Cover annealing furnaces for use with strip and sheet (*see* fig. 242).
- (c) Bogie furnaces for heat treatment of forgings.



*Fig. 242. Radiant tube type annealing furnace used for strip or sheet (after H. Edwards & F. R. Jones).*

The conditions in these furnaces are less severe than in the reheating furnace or soaking pit and, therefore, do not present any particular problem as regards refractories. They are, however, worthy of study, in that use can be made in many designs either of high porosity refractories or of genuine insulating bricks, and substantial economies made both in engineering costs and in fuel or electric power requirements.







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## Chapter XIV

# THE CASTING PIT

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THIS SUBJECT SHOULD logically be discussed as Chapter XIII, since the steel must be cast before it can be reheated for rolling or forging. It has only been placed at the end so that the sections dealing with furnaces could be kept consecutive. The casting pit (usually referred to in the U.S.A. as a pouring pit) not only requires the use of refractory lined ladles with their ancillary nozzles, stoppers and ladle sleeves, but also large quantities of refractories for mould tops and bottom pouring assemblies. The problems presented to the refractories technologist are unusual, both because the short contact time prevents equilibrium conditions being established and because any failure in the refractories, whether by spalling or slagging, may lead to dirty steel and a consequent waste of material, including all the refractories used in the previous processes.

The short contact times and the consequent steep temperature gradients lead to many anomalies, *e.g.*, the excellent results obtained, particularly in the United States, with bricks whose melting point is scarcely high enough to warrant their description as refractories. Similarly in the bottom pouring assembly the use of relatively low melting point material may well lead to considerable slag attack, but if the reaction product is really fluid it may well rise to the surface and give cleaner steel than where a more refractory brick is employed.

Such anomalies make specifications difficult if not actually undesirable and justify the present approach, in which the properties of new materials are compared with those of products that have already proved their usefulness. A good example of this latter procedure was provided by a major failure that occurred when making forging ingots, several of which were found to contain pieces of brick of up to 1 inch in diameter. Laboratory data and in particular determinations of X-ray diffraction spectra, porosity and permeability, showed that the probable cause of the trouble was low thermal shock resistance, which in turn appeared to be due to a surprising increase in apparent firing temperature from 1250° to 1400°C. It was subsequently found that the bricks had been fired at this temperature in order to meet the

requirements of other materials set lower in the kiln, and that as a result the particular mould top bricks were quite useless for the purpose for which they were intended.

The life of ladles varies greatly from one plant to another but some idea of the costs involved can be gathered from the fact that in a plant where the life is 10 heats, and the ladle capacity 100 tons, the refractories +labour cost is approximately 2s. per ton. Where bottom pouring is employed additional costs are involved—possibly 4s. per ton, a figure that can only be justified by the improved quality of the ingot.

## LADLES

When the taphole has been opened, either by the traditional hammer and bar followed by oxygen lance, or by the new method of jet tapping, where an explosive charge is used to achieve final penetration, the steel rushes down the lander into the ladle, providing one of the most spectacular sights in the steel industry (fig. 243, page 587).

### (a) CONSTRUCTION

The precise design of bricking in the ladle varies greatly from plant to plant, but the general arrangement in large ladles is similar throughout the world and is well represented by fig. 244, reproduced from the A.S.T.M. Manual on Refractory Materials. Small ladles, such as are employed in foundries, are frequently lip-poured, but most steelplant ladles are fitted either with a single or double stopper rod and nozzle arrangement. This permits of a precisely located vertical stream, which can be shut off when the ladle is transferred from one tundish, or mould, to another, even though in practice it is often kept at least partly open while the transfer occurs.

The thickness of the lining used varies with the ladle size, but is rarely less than a  $4\frac{1}{2}$  in. inner lining with a 2 in. safety course behind. In the bottom section of the ladle, where the time of contact with metal and slag is longer, a thicker lining is normally employed, for example 6 in. blocks instead of the  $4\frac{1}{2}$  in. lining used above. Even this thickness would prove inadequate if equilibrium were reached but in fact it is rarely approached, since the steel is normally teemed within 20 to 40 minutes of being tapped into the ladle. The fact that the penetration of temperature (see fig. 245) is slow and that a linear gradient is not even reached in the full time during which steel remains in the ladle, explains in part why relatively non-refractory bricks can give good results. In many ladles extra thickness is provided in the portion of the side-wall opposite to the furnace spout in order to compensate

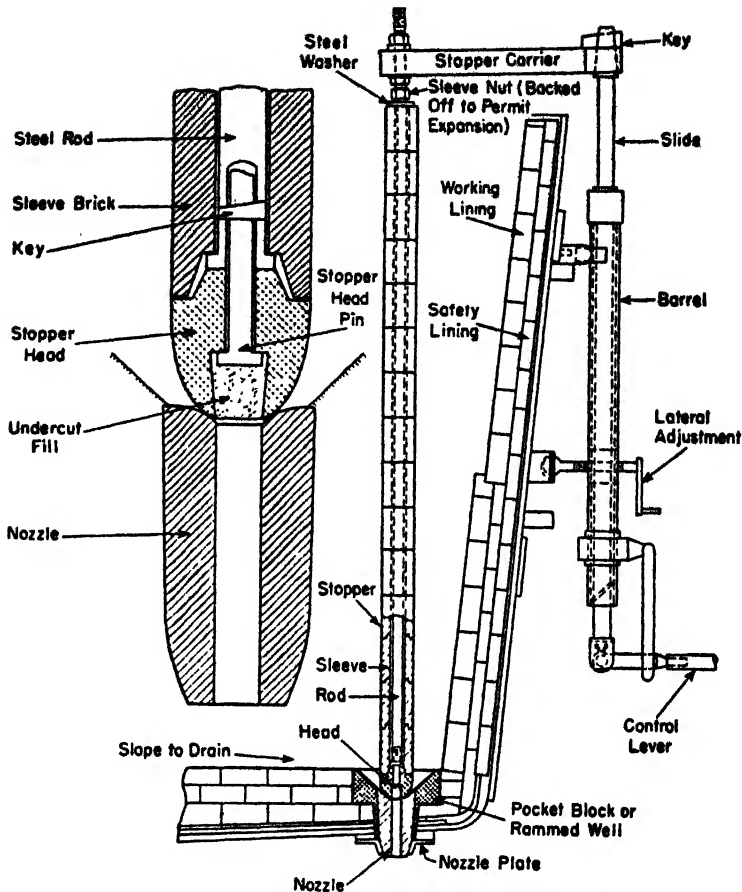


Fig. 244. Arrangement of ladle refractories (after ASTM Standards: 1952).

for the additional erosion caused by the impacting stream. This thicker area is frequently referred to as the "striker patch".

Since steel tightness is vital on grounds of safety and economy, the joints in a ladle are of great importance. Accuracy of shape and size are therefore a first essential in ladle bricks. Where the standard is really excellent, bricks may even be laid dry, though in general it is preferable to use at least dipped joints. The quality of the cement employed is important, particularly if the bricks themselves do not show a high after expansion. The inner lining frequently consists of a series of rings, each built of soldier courses, *i.e.*, 9 in. brick standing on

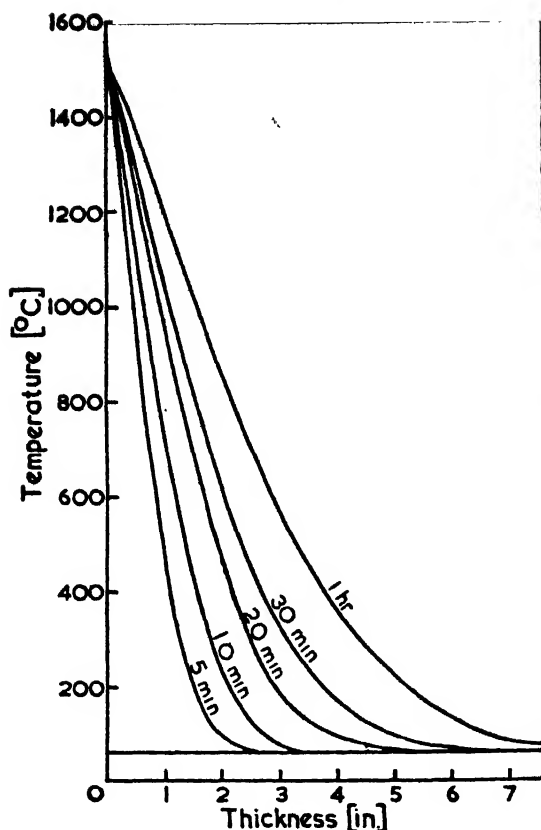


Fig. 245. Variation with time of temperature gradient in  $7\frac{1}{2}$  in. thick ladle lining. Initial temperature of steel  $1585^{\circ}\text{C}$ . (after Sosman).

end. An alternative method, which it is claimed gives a more satisfactory lining because it minimises the amount of "piecing-up", is that in which the bricks are built in a spiral. The only difficulty in such bricking lies in the first ring, which must taper from zero to single brick height.

In foundries, and to a less extent in electric steelplants, substantial use is made of monolithic ladles made by ramming sand-clay or fireclay grog—clay mixtures. On the Continent this type of construction is used with ladles of even 80-ton capacity. That such ladles can give a good life, particularly if frequently parged, has been well demonstrated, but whether on an overall basis they can be considered satisfactory where good bricks are available at reasonable prices is far from proved. An

argument frequently raised against their use is the risk of inclusions, but recent work suggests that steel contamination is only likely to come from the area immediately adjacent to the nozzle.

Before a ladle (whether bricked or monolithic) goes into service, adequate drying is essential. The normal method is to heat up the ladle to red heat, by means of coke braziers, or gas flares, but there is still great variability from plant to plant in the duration of this heating and the maximum temperature attained. What is clearly important is that there must be no risk of sudden steam formation when the metal enters the ladle, since this might well lead to an explosion, or at least to premature failure of the ladle lining.

#### (b) MATERIALS

A vast amount of research has been done on this subject in recent years. Those who are directly concerned are, therefore, recommended to make a detailed study of the literature, starting for example with the Symposium on Casting Pit Refractories, and in particular Ladle Linings, published in the October, 1954, issue of the Transactions of the British Ceramic Society.

#### *Fireclay brick*

One firm conclusion arising from all the above work is that no matter what quality of firebrick is employed—and virtually all steel ladles are lined with fireclay bricks—density is of paramount importance, anything that can be done to increase it normally resulting in improved ladle life.

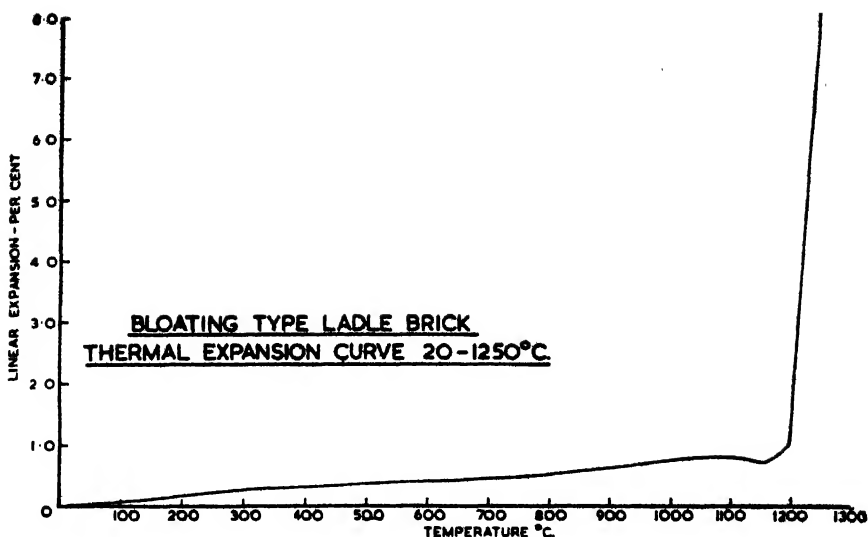
The bricks employed still fall into 2 main categories:

- (1) The siliceous type fireclay brick, having a relatively low melting point but also high density, low permeability and a tendency to bloat on refiring.
- (2) The medium alumina (35 to 42 per cent.) type brick, having a refractoriness of, say, 1650°C. or over, and in general a rather higher porosity than the siliceous or so-called "potty" type.

The properties of a number of British bricks of both types, and also of two outstanding bricks used in the United States and Germany respectively, are shown in Table LX. It will be seen from this that the American brick has an even lower melting point (1530°C.) than the low alumina British types and a remarkable bloat—42.1 per cent. linear after 2 hours at 1410°C. The extraordinary bloating properties of this particular brick are well illustrated by fig. 246, the volume of the sample fired for 2 hours at 1410°C. being more than twice the initial

TABLE LX  
PROPERTIES OF LADLE BRICKS

Code No.	British low alumina type		British medium alumina type				American low alumina type	German low alumina type
	F.17	F.34	F.12	F.2	F.32	F.33		
Apparent porosity, per cent. . .	23.7	18.7	18.2	21.7	13.7	17.0	12.8	9.3
Bulk density, g.p.ml. . .	2.00	2.05	2.11	1.98	2.00	2.17	2.26	2.21
Cold crushing strength, lb. per sq. in. (on end) . . . . .	6910	3140	—	4140	3530	4190	4720	—
Permeability to air, c.g.s. units, Perp. 9 × 3 in. face (1 skin) . .	0.0036	0.0036	0.0084	0.013	0.014	0.046	0.005	0.004
Permanent linear change on re- heating, 2 hrs. @ 1410°C. . .	+3.6	+3.6	-0.7	-0.6	-1.4	-0.3	+42.1	+1.1
Refractoriness-under-load, 50 lb. per sq. in.: °C.	1010	1100	1350	1220	1150	1300	—	—
Initial softening . . . . .	1420	1380	1440	1450	1400	1440	—	—
Rapid softening . . . . .	1520	1520	1580	1540	1550	1600	—	—
Fail or 10 per cent. collapse . .	1550	1580	1700	1680	1710	1710	1530	1610
Refractoriness, °C. . . . .	12	19	13	30+	30+	30+	11	8
Thermal shock resistance . . . .								



*Fig. 246. Sudden bloat in American ladle brick on approaching 1200°C.*

value. There is little doubt that it is this combination of high initial density, low permeability and perfection of shape combined with the substantial bloat, which in service results in tight joints and an even more vitrified working face, that explains the excellent service generally reported with this product. The German brick shows the same high bulk density and low permeability but a very much smaller bloating tendency and rather higher refractoriness. Incidentally, it is interesting to note that all these bricks have a thermal shock resistance of at least 8 reversals and at this value show no signs of spalling in service.

The mechanism responsible for the remarkable bloat shown by the American brick is still not completely understood, though it has been suggested that it is due to the evolution of sulphurous gases at a stage when the brick has already softened and the pores have consequently become sealed. The softening itself is doubtless associated with an alkali content of over 3 per cent. together with the siliceous character of the brick—alumina 23.5 per cent. and silica 67.5 per cent. Several workers have attempted to produce bricks of this type, *e.g.*, by the addition of sulphate compounds, and with some measure of success, though there is little doubt that the task of producing a good ladle brick is in this case greatly simplified by the character of the clays employed.

The relative importance of different properties in ladle bricks still requires a great deal of study and is unlikely to be elucidated merely by



comparing commercial bricks of known properties. More promising is the production in quantities sufficient at least for panel trials of a range of bricks made from each of a number of clays, in which the physical properties are deliberately modified, *e.g.*, by fineness of grinding, additions, and method of manufacture, including firing treatment. Given such tests it may be possible to show just how much bloating is necessary to give optimum results and what further gains may be expected from decreases in porosity and permeability. Such tests may well go hand in hand with dip tests, of the type described by Rousseau and Mackenzie, in which bars cut from brick are dipped into a bath of steel covered with basic slag. Provided there is adequate temperature control and the atmosphere is such as to maintain the basic slag in the condition in which it exists on the ladle, quick results obtained in this way may well tie up satisfactorily with those obtained in actual ladles, and thus speed up the development process. Fig. 247, p. 588, shows a typical result of such a test, the brick on the left, which shows the small cut, being the high grade German brick referred to above, whilst the brick on the right is one of the less successful British products of the 40 per cent. alumina type and relatively low density. Fig. 248, p. 589, shows broken sections of two bars after such a dip test, that on the left being a German brick of high bulk density but relative volume stability, and that on the right, being a less refractory American brick, possessing a marked bloating tendency.

Where bricks show a marked bloating tendency and are of excellent shape, ladles may even be built dry but in general the use of a good quality cement is desirable and for this purpose materials high in quartz and, having, therefore, a high after-expansion are often used, since they help to give a steel tight ladle. The thickness of joint is mainly a function of brick shape, being very thin where the latter is excellent and thicker where unevenness in bricks must be accommodated.

#### *Plumbago (clay-graphite) brick*

Trials made both in Great Britain (fig. 249, p. 589) and more recently in the U.S.S.R., indicate that plumbago bricks withstand conditions in steelworks ladles far better than the normal fireclay product. Thus Budnikov found that plumbago bricks, containing 30 per cent. graphite, gave lives of 16 to 19 casts on manganese steel compared with 5 to 6 casts on normal firebrick lining. This result would be entirely satisfactory were it not for the high cost of plumbago bricks, which in turn is due to the presence of 30 per cent. or more of flake graphite. Studies of their other properties show that they are not usually particularly dense

(25 to 33 per cent. porosity, bulk density 1.7 to 1.8 g.p.ml.) but that they have a low permeability. Their main advantage is their high slag resistance which is due to the presence of the graphite. Just how much of the resistance is physical and how much chemical still remains to be shown. It is well known, for example from crucible studies, that graphite flakes obstruct slag penetration and that for this reason even their orientation is important. It may well be that the presence of carbon also has an effect on the specific corrosiveness of the slag. The major difficulty with the use of such bricks is the production of a cement that will stand up to attack as well as the bricks themselves. In most of the trials so far carried out attack between the bricks is severe and the ladle soon assumes a cobblestone appearance and has to come off not because the average thickness is low but because there is risk of penetration between the bricks. Incidentally the use of cements high in graphite for patching has been found to lead to quite dangerous frothing of slags, presumably due to the reduction of iron oxide by carbon and the consequent release of large quantities of carbon monoxide.

#### *Rammed ladles*

Considerable use has been made on the Continent of ladles rammed from natural bonded sands, *e.g.*, those found in Belgium. Some of these sands are not particularly refractory, having a melting point of the order of 1670°C., due to the presence of over 5 per cent. of alumina. They have a medium grain size distribution, with about 5 per cent. on 22 mesh, 50 per cent. between 22 to 72 mesh and 45 per cent. less than 72 mesh, of which 20 per cent. passes 150 mesh. For ramming purposes they are used with a moisture content of approximately 8 to 10 per cent, the ladle being dried out in the ordinary way. It is claimed that under certain conditions such linings last twice as long as firebrick linings, but such statements require careful qualification in terms of the relative amounts of repair work involved. Given a satisfactory patching material, it is clear that such a ladle could be kept in service almost indefinitely, but the economics of repeated patching are by no means certain. Trials of such sands have been made in Great Britain—mainly in foundries but also in ladles of up to 80-ton capacity—but the results obtained have not led to their widespread use. Their advantages are, however, quite clear: joints are eliminated, the siliceous batch having a positive expansion on firing shows a minimum of cracking, whilst the relatively non-refractory material glazes quite readily.

#### *Basic ladles*

Considerable research has been done, particularly in Great Britain, on the production of satisfactory basic ladle bricks. The principal

objective in this work was metallurgical, *viz.*, to reduce the tendency to rephosphorisation during the last stages of teeming. Since some of the latest studies cast some doubt on whether basic ladles as now lined would in fact achieve this objective, the main stress in future is likely to be on the production of a basic ladle brick that will justify itself purely on a refractory cost basis. This target is a difficult one to achieve with the present cost ratio of basic to fireclay bricks.

Numerous materials have been tested, starting with ordinary tarred doloma, and proceeding through cold setting stabilised dolomite cements and special types of unfired stabilised dolomite bricks to a variety of serpentine-clay and serpentine-magnesite bricks. The results obtained by different workers all show the same general character, *viz.*, an overall life greater than fireclay but not sufficiently so to warrant the higher initial costs, and a build up of slag and metal on the working face (*see* fig. 250, p. 590) which gradually reduces the ladle capacity to a point where stripping becomes essential. The first removal of slag and metal although scarcely convenient is by no means impossible, but repeated removal leads to damage to the bricks and reveals weaknesses, such as metal in the joints, that soon causes a loss of faith in the safeness of the lining. The idea that the build up is due to the higher thermal conductivity of basic bricks would not appear to fit the facts, since measurements made on unfired bricks show conductivities of the same order as those of fireclay bricks. The heat capacity on the other hand is substantially greater and some extra chill may, therefore, be expected. What is probably more important, however, is the fact that attack by basic slag on a fireclay brick yields a product having a lower melting point than either brick or slag, whereas attack by slag on basic bricks immediately thickens the slag and in consequence is likely to result in adherence.

#### (c) LIFE AND CAUSES OF FAILURE

The life even of straight fireclay brick linings varies greatly from plant to plant, being as low as 3 heats for low carbon rimming steel, where the slag may contain as much as 25 per cent. iron oxide, to more than 30 heats in plants operating tilting furnaces on medium carbon qualities. In the latter the ladles carry very little slag cover. Typical practice for, say, 100-ton ladles is approximately 8 to 15 heats, though even in a shop with an average life of, say, 10 heats, there is likely to be a variation, at least from 6 to 14 heats in ladle lives obtained over, say, a month's operation. The general cause of failure is solution of the bricks by the slag, but linings can also be taken off for other reasons, *e.g.*, bad joints, or localised attack. It is interesting to note that although

the thickness of the inner lining is generally  $4\frac{1}{2}$  in. only about one-third of the brick weight is actually used, the remainder being thrown away.

Numerous investigations have been carried out during recent years on the causes of wear on ladle linings, one of the most detailed being that by Dodd and Green, published in 1946. They show that even within a shop the operating conditions vary enormously, *e.g.*, temperature may change by as much as  $100^{\circ}\text{C}$ ., the time of contact vary from  $\frac{1}{2}$  to  $1\frac{1}{2}$  hours, the weight of slag from  $\frac{1}{2}$  to 12 tons and the slag composition over the range: CaO 37 to 47 per cent., FeO 9 to 33 per cent. and  $\text{SiO}_2$  7 to 16 per cent.

They consider that the volume of the slag carried is the most important single factor, a conclusion that has been amply confirmed by later workers. Within the limits studied, temperature variations seem to have little effect but basicity of slag they consider to be an important factor in controlling the rate of solution. They attribute the corrosive action of ferruginous slags to their tendency to give a more fluid slag that penetrates more deeply into the refractory. They also point out the danger of under-burned ladle bricks, to which might be added the danger of badly burned ladle bricks containing carbonaceous "black-hearts", which experience shows are particularly susceptible to attack yielding the localised wear illustrated in fig. 251, p. 590.

The desirability of minimising the amount of slag in the ladle is particularly stressed by Andrew in his statistical investigation into the factors affecting the life of ladle linings. The great variations found in slag weight are attributed in the main to the uncertainty of the amount of steel present in the furnace, which necessitates the use of a far larger ladle than would otherwise be necessary. This in turn leads to a high average slag depth and, therefore, also to serious wear. It is quite possible that the improved ladle life resulting from better control of charge weight would be itself sufficient to pay for the weighing.

Van Gijn has suggested an ingenious way of dealing with the increased slag depth normally associated with ladle wear. In his paper on the performance of Continental ladle and runner bricks, he recommends the use of a special ladle spout filled with ladle bricks retained by removable iron bars. After about two casts in one particular plant a layer of bricks and the corresponding bar are removed, this process being repeated at intervals. By this simple means the average life of ladles in a particular plant was increased from 13 to 15 heats, whilst in subsequent work a gain of over twice this amount was obtained.

The relation of ladle life to brick quality is still far from clear apart from the fact that high density is of paramount importance. L. Halm reports that her co-worker P. Lapoujade has developed a quality index

based on one previously suggested by Debenham and Eusner. This is as follows:

$$I = 2 \left( \frac{P}{D} + 100p \right) + \frac{10,000}{2R} \pm 2V$$

where:  $I$  = Quality index

$P$  = Apparent porosity (%)

$D$  = Bulk density ( $\text{g}/\text{cm}^3$ )

$p$  = Permeability  $\text{cm}^3/\text{cm}/\text{cm}^2/\text{sec}^{-1}/(\text{g}/\text{cm}^3)^{-1}$

$R$  = Cold crushing-strength ( $\text{kg}/\text{cm}^2$ )

$V$  = Permanent linear change (%) after refiring for 2 hr. at  $1500^\circ\text{C}$ . The last term in the formula is positive when the change is a contraction and negative when it is an expansion, because the latter is desirable.

Such formulae are wide open to criticism on the grounds that one cannot add dissimilar quantities, such as permeability and the reciprocal of cold crushing strength but they may nevertheless represent numerically an approximation to the factors controlling ladle life.

More recently attempts have been made to develop comparative tests that would enable the probable life of a new brick in comparison with one already employed to be predicted. Thus Rousseau developed a technique in which samples of brick were plunged simultaneously into a bath of molten steel, or of fluid slag of suitable basicity, and the loss in weight or volume taken as a measure of the probable attack. The later work by Mackenzie, in which rods of brick are dipped into molten steel and slag, has already been mentioned.

General loss of thickness is, however, by no means the sole cause for ultimate ladle failure. There may be localised attack at the joints due for example to poorly shaped bricks, after-contraction, or low grade cement, or the localised attack within bricks mentioned above as due to black-hearts. In addition improperly dried ladles may result in localised disruption, or even to an explosion, whilst cold metal or long casting periods may cause heavy skulls and consequent damage to linings during their removal. These latter occur quite frequently on ladles where the slag cover is small and heat losses therefore rather high.

#### (d) LINES OF IMPROVEMENT

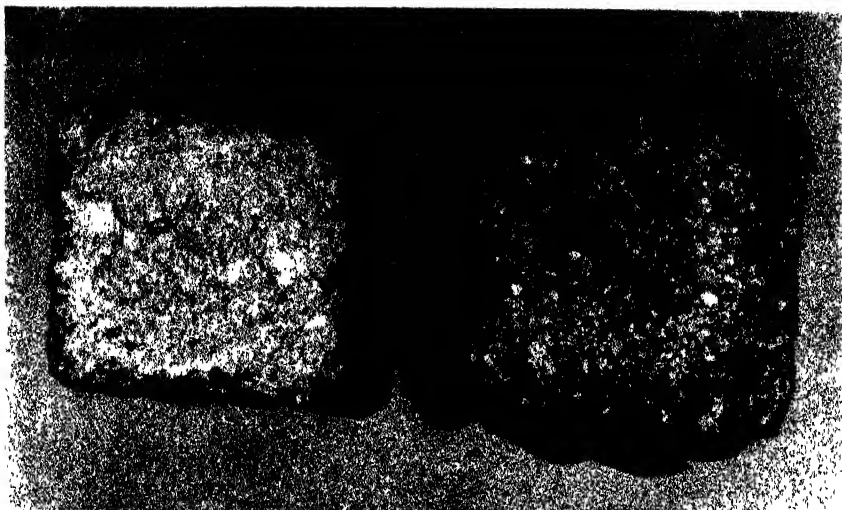
Of the possible future developments, the production of even denser fireclay bricks would seem to be the most profitable: a drop in porosity of, say, 10 per cent. leading to an improvement of as much as 20 per cent. in ladle life. If such high densities and vitrifying tendencies can be produced in more aluminous materials even greater lives may be



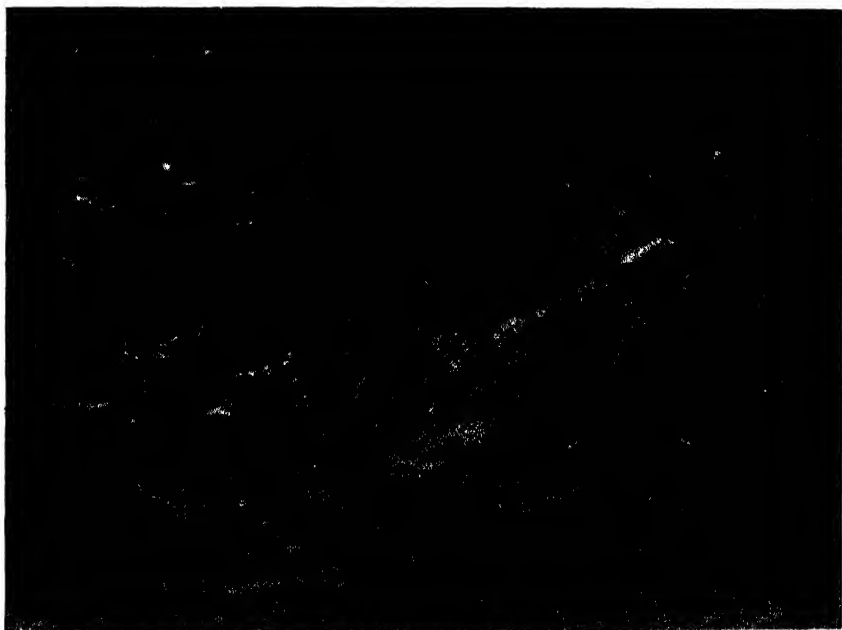
*Fig. 243. Open-hearth furnace tapping into ladle.*



*Fig. 247. Dip test on dense (left) and porous (right) ladle brick testpieces.*

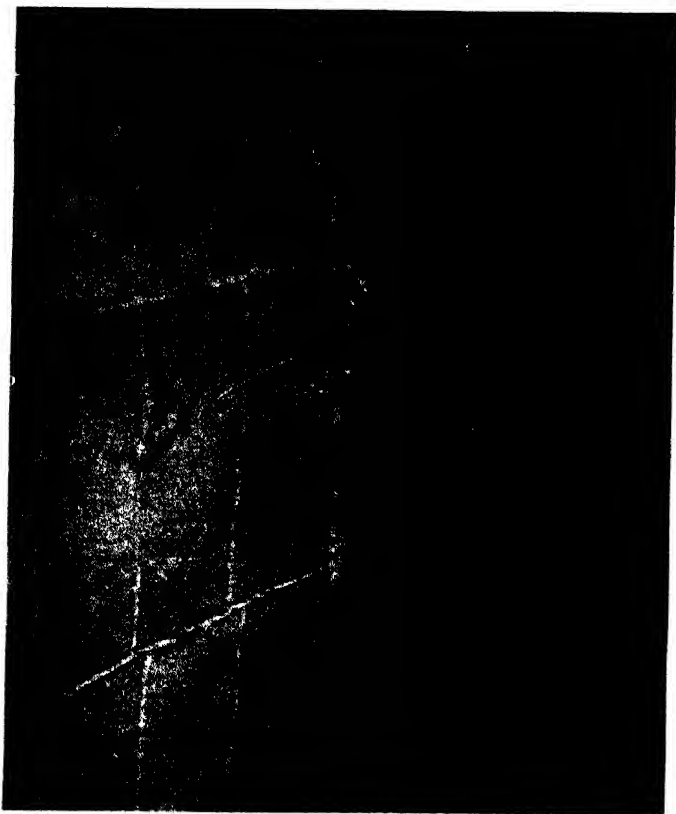


*Fig. 248. Cross-section through dip test rods: left, non-bloating; right, bloating type.*



*Fig. 249. Panel of plumbago (clay-graphite) bricks in the side of medium alumina fireclay brick ladle. Plumbago bricks show little wear, although surrounding bricks have gone back by almost their full thickness.*





*Fig. 250. Slag build-up on surface of stabilised dolomite ladle bricks.*



*Fig. 251. Cored (black-hearted) ladle brick showing abnormal wear in the cored portion.*

expected, but present work indicates that the use of relatively siliceous (25 per cent. alumina) clays of relatively low iron content are likely to achieve success more readily.

Both plumbago and basic bricks are still found far more inherently durable than fireclay linings, but are still subject to troubles, notably attack between the bricks with plumbago, and slag adherence and subsequent disruption with basic linings. With both types of refractory there is a potential gain provided these limitations can be overcome, but sufficient work has been done to show that the solution is not likely to be an easy one. As regards basic linings the successful product might well arise from a complete break with present ideas, *e.g.*, the development of, say, a special magnesia-alumina-silica clinker, whose properties were such that build up did not occur but wear was unusually slow.

## NOZZLES AND STOPPERS

The job specification for nozzles and stoppers has been neatly described by Dodd and Green in the Iron and Steel Institute Special Report No. 26 as follows: "The perfect nozzle would permit the teeming of the entire cast of steel at a constant rate without splashing on the sides of the ingot mould, and without contaminating the metal; the nozzle would also maintain a perfect seating for the stopper head so that no dribbling would occur while the ladle was being moved from one mould to the next". Fig. 252, p. 595, shows teeming in progress.

Ekholm and Hower in their very entertaining report to the A.I.M.E. on nozzle and stopper head properties suggest a similar specification but go on to point out that leaking or running stoppers may be caused by improper assembly or by faulty installation and drying of the nozzle, as well as by limitations of the nozzles and stoppers themselves. They point out that when the mould is full and the stopper is lowered a good closure will be obtained if the relative contours of the head and nozzle have remained constant or the stopper head is relatively hard compared with the nozzle and can, therefore, deform the latter to make a perfect closure. If such a closure is not obtained and dribbling occurs then a groove may soon be cut in the nozzle that will make a further clean shut-off impossible.

Three main methods exist for attaching the stopper end to the mild steel stopper rod:

- (1) The rod may be threaded and the nozzle screwed on to the end, the hole in the stopper going either part or all the way through.
- (2) The stopper may be attached to the rod by means of a stopper head pin and key, as shown in fig. 244.

(3) The stopper may be held on to a threaded rod by means of a nut.

Needless to say where a hole initially exists right through the stopper, the open end must be blocked with suitable cement prior to use. The rod itself is of course protected by so-called rod covers or ladle sleeves, the properties of which will be discussed in a later section.

Study of the properties of nozzles and stoppers shows a marked contrast between British and American practice. As will be seen from Table LXI, there is little difference between British nozzles and stoppers made by the same firm. Indeed no great difference was expected since they are made from similar clay batches and are fired in the same kilns. In the United States on the other hand the general custom is to use a relatively non-refractory nozzle with a clay-graphite type stopper. This material tends to be relatively hard and tough at high temperatures even though the clays used are not particularly high in alumina. The American practice would appear to be more logical, in that it is easier to see how a good seal can be obtained by pushing a hard stopper into a relatively soft clay nozzle.

The success of British clay stoppers is doubtless due to the care given both in raw materials and in manufacture both to these and to the corresponding nozzles. As will be observed from Table LXI, the clays used are of the 39 per cent. alumina type, and the procedure is such as to yield a dense product, of extremely low permeability. As will be seen from data given in the same table, American nozzles may also be of high bulk density but have a relatively low refractoriness—only 1550°C. for the sample examined. They have, however, in addition a marked bloating tendency—volume expansions of up to 75 per cent. have been obtained on refiring, indicating plasticity at the working temperature. It has been suggested that this property is valuable not only because it enables a good seating to be obtained by the stopper end but also because any cracks formed tend to weld up rather than cut out as they are likely to do with more refractory products.

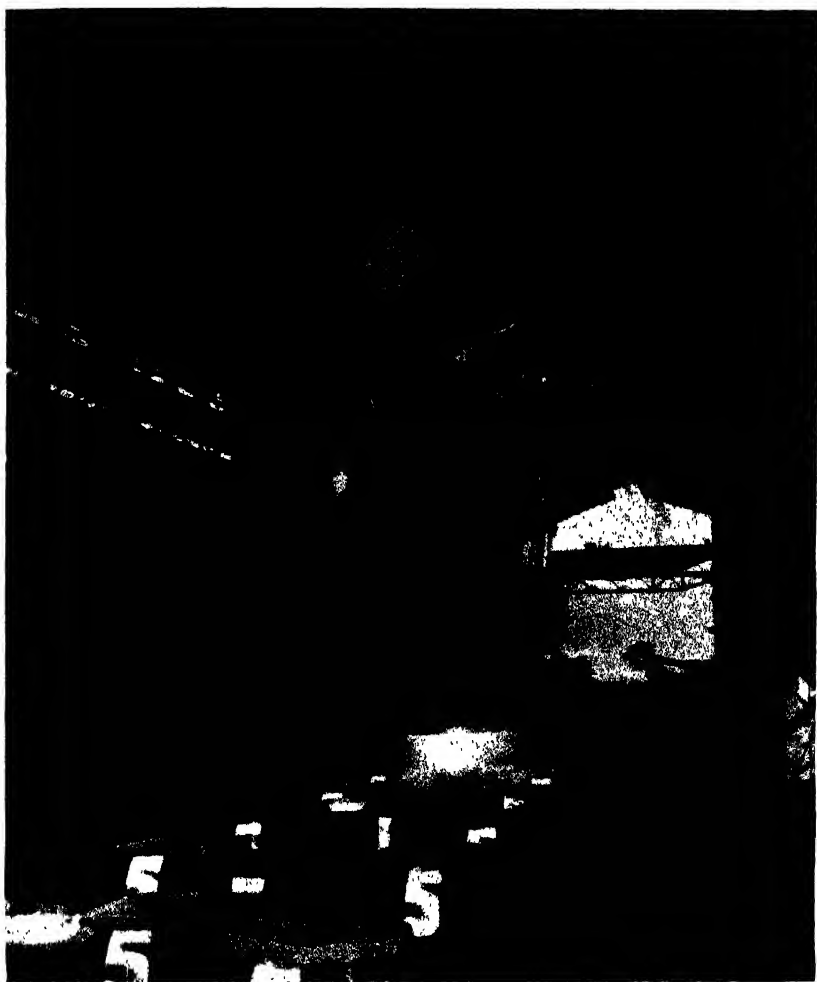
Comparatively little data is available regarding the properties of clay-graphite stoppers, though L. Halm in a recent paper gives the data reproduced in Table LXII. It will be seen that the samples examined contain between 10 and 18 per cent. carbon, *i.e.*, roughly half that found in the plumbago ladle bricks. Both general experience, *e.g.*, with steel melting crucibles, and direct laboratory tests, have shown that in some peculiar way carbon yields good strength with such refractories at high temperatures, even where, as in the present case, the alumina content of the clay is not particularly high. It is interesting to note that the porosities of the particular stoppers are quite high, though it will be seen that the best results were obtained with the

TABLE LXI  
PROPERTIES OF NOZZLES AND STOPPERS

	British nozzles		British stoppers		U.S.A. nozzle
	A	B	A	B	
Apparent porosity, per cent.	21.3	25.2	16.4	16.9	16.0
Bulk density, g.p.ml.	2.09	2.00	2.17	2.14	2.14
Apparent solid density, g.p.ml.	2.65	2.67	2.59	2.58	2.55
Permeability to air, c.g.s. units (1 skin)	0.0004	0.0034	0.0003	0.0015	—
Permanent linear change on reheating, 2 hrs. at 1410°C. . .	+5.3	+0.7	+6.4	+7.0	ca. +25.0
Refractoriness-under-load (50 lb. per sq. in.): °C.					
Initial softening . . .	1130	1200	1110	1100	
Rapid softening . . .	1410	1330	1380	1300	
10 per cent. deformation . . .	1480	1520	1520	1470	
Thermal shock resistance (900°C. test) (3 in. × 2 in. × 2 in. section) . . .	4.5	30+, 11	25, 27	30+, 21	
Refractoriness, °C. . .	avg. 5	avg. 21+	avg. 26	avg. 26+	
Free quartz (per cent.): estimated by microscope	1700	1730	1730	1720	1550
Chemical analysis (per cent.):					
SiO <sub>2</sub> . . .	4	2	4	2	—
FeO . . .	54.26	54.61	54.34	53.31	
Al <sub>2</sub> O <sub>3</sub> . . .	3.20 (Fe <sub>2</sub> O <sub>3</sub> )	2.18	3.76 (Fe <sub>2</sub> O <sub>3</sub> )	2.70	
TiO <sub>2</sub> . . .	38.82	38.99	38.80	39.68	
CaO . . .	1.26	1.40	1.18	1.50	
MgO . . .	0.20	0.04	0.52	0.04	
Loss on ignition	0.94	0.83	1.02	1.10	
	0.26	—	0.32	—	

TABLE LXII  
 PROPERTIES OF SOME CLAY-GRAPHITE STOPPERS—OPEN-HEARTH FURNACE STEEL  
 (after L. Halm)

	Make			
	TA	TB	TC	TD
Chemical analysis (per cent.): Carbon ..	13.23	18.33	15.04	10.47
SiO <sub>2</sub> ..	52.01	51.05	44.22	47.15
TiO <sub>2</sub> ..	1.08	1.12	1.37	1.86
Al <sub>2</sub> O <sub>3</sub> ..	29.48	26.32	36.73	36.96
Fe <sub>2</sub> O <sub>3</sub> ..	2.06	1.52	1.78	2.52
CaO ..	0.88	0.14	0.31	0.94
MgO ..	0.11	trace	trace	trace
Na <sub>2</sub> O + K <sub>2</sub> O ..	0.81	1.45	0.91	0.19
Bulk density (g/cm <sup>3</sup> ) ..	1.96	1.89	1.86	1.83
Apparent porosity (per cent.) ..	21.5	24	28.5	32
Refractoriness under load (°C.) (French standard)				
Commencement of subsidence ..	1340°	1380°	1320°	1340°
0.5 per cent. " ..	1390°	1435°	1375°	1420°
5 per cent. " ..	1490°	1535°	1465°	1480°
10 per cent. " ..	1525°	1570°	1525°	1530°

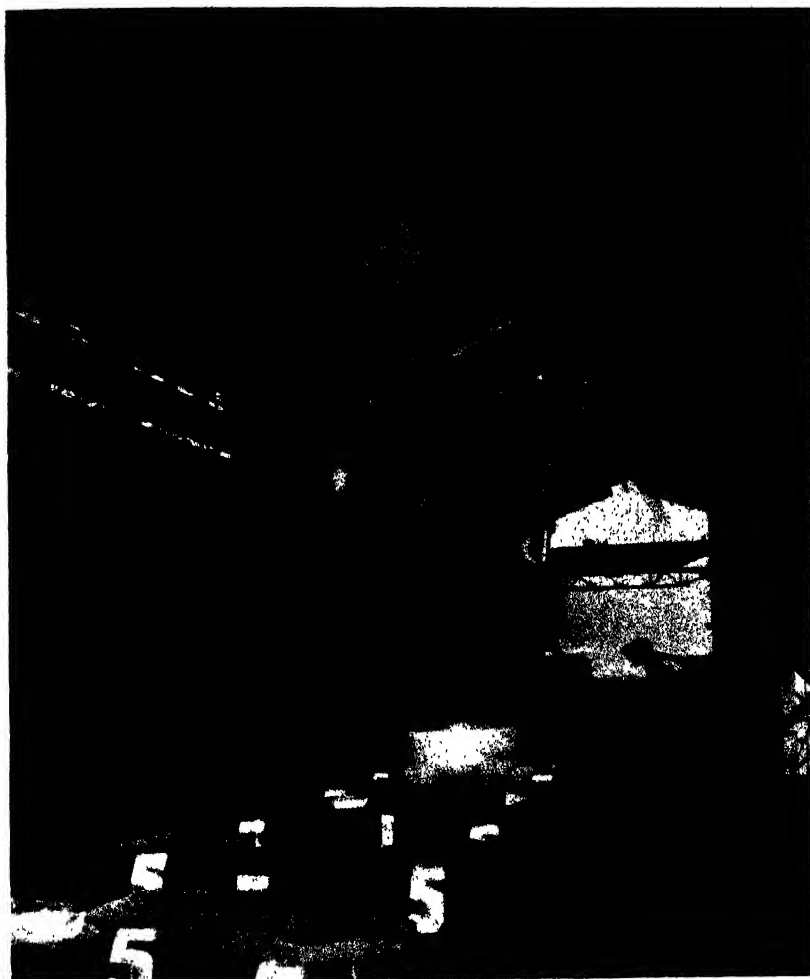


*Fig. 252. 80-ton ladle teeming into the trumpet of a bottom pouring set-up.*

TABLE LXII

PROPERTIES OF SOME CLAY-GRAPHITE STOPPERS—OPEN-HEARTH FURNACE STEEL  
(after L. Halm)

	Make			
	TA	TB	TC	TD
Chemical analysis (per cent.): Carbon	13.23	18.33	15.04	10.47
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Al <sub>2</sub> O <sub>3</sub>	29.48	26.32	36.73	36.96
Fe <sub>2</sub> O <sub>3</sub>	2.06	1.52	1.78	2.52
CaO	0.88	0.14	0.31	0.94
MgO	0.11	trace	trace	trace
Na <sub>2</sub> O + K <sub>2</sub> O	0.81	1.45	0.91	0.19
Bulk density (g/cm <sup>3</sup> )	1.96	1.89	1.86	1.83
Apparent porosity (per cent.)	21.5	24	28.5	32
Refractoriness under load (°C.) (French standard)				
Commencement of subsidence	1340°	1380°	1320°	1340°
0.5 per cent. "	1390°	1435°	1375°	1420°
5 per cent. "	1490°	1535°	1465°	1480°
10 per cent. "	1525°	1570°	1525°	1530°



*Fig. 252. 80-ton ladle teeming into the trumpet of a bottom pouring set-up.*





*Fig. 256. Fireclay nozzle, stopper and rod cover for an 80-ton ladle.*

denser products. The refractoriness-under-load results, which it should be noted were obtained by the French standard method, are not particularly high when compared with those of fireclay nozzles similarly tested, but some lowering may have occurred due to burning out of the graphite during the test.

A property of the nozzle which greatly concerns those working in the casting-pit is the rate at which it erodes, since this determines the speed with which the steel is teemed into the ingot moulds. If, as is generally assumed, a steady pouring rate is ideal, then some erosion of the nozzle is desirable in order to compensate for the gradual fall in ferrostatic head in the ladle. Fig. 253, shows a series of curves for different

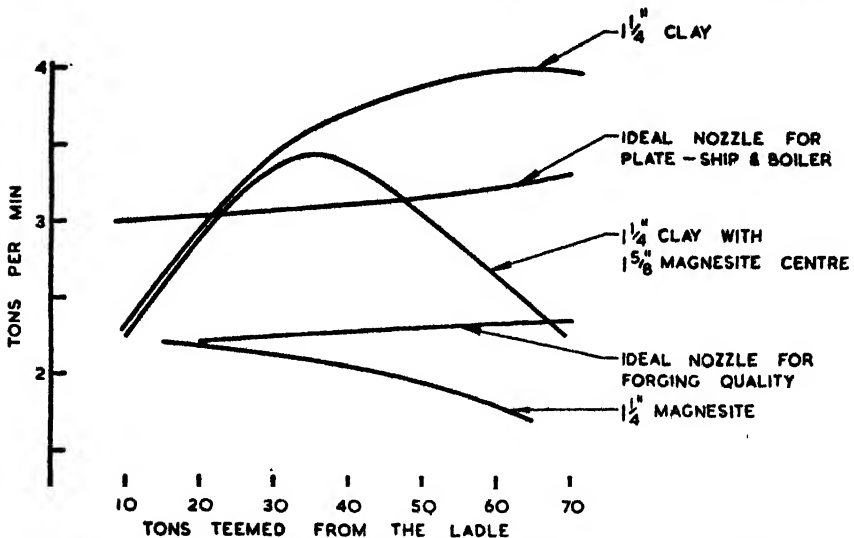


Fig. 253. Influence of nozzle type on rate of teeming various steels (A. Jackson).

types of nozzles reported by A. Jackson, from which it will be seen that the increase in pouring speed, due to the use of a clay nozzle, can be turned into a decrease by using a magnesite nozzle, or an intermediate curve by the use of a magnesite insert, different types of which are shown in fig. 254. Ekholm and Hower have given curves (see fig. 255) for the erosion in inches on a clay nozzle when pouring plain carbon and high manganese-sulphur steel. This information is in line with general experience, and in particular with the reports by Snow and Schea, who found that with aluminium killed steels the amount of erosion decreased as the refractoriness of the nozzle increased, but that with high manganese steels the erosion was worst with the more refractory nozzles, presumably due to the greater porosity and the ease with

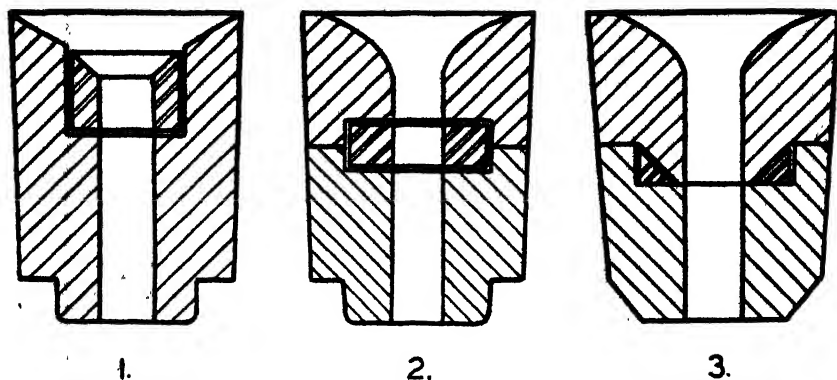


Fig. 254. Three types of fireclay nozzle with magnesite inserts.

which FeO and MnO bearing liquids were able to penetrate and, therefore, soften the surface layers. They suggest that less refractory—bloating type—nozzles form a relatively impervious layer and reduce such penetration. Ekholm and Hower also find that nozzles of greater bulk density show a lower erosion rate than those of lower bulk density.

A major difficulty in casting steel is that such things as temperature and aluminium addition have a marked effect on fluidity and that precise pouring rates are not always easy to predict. It would clearly

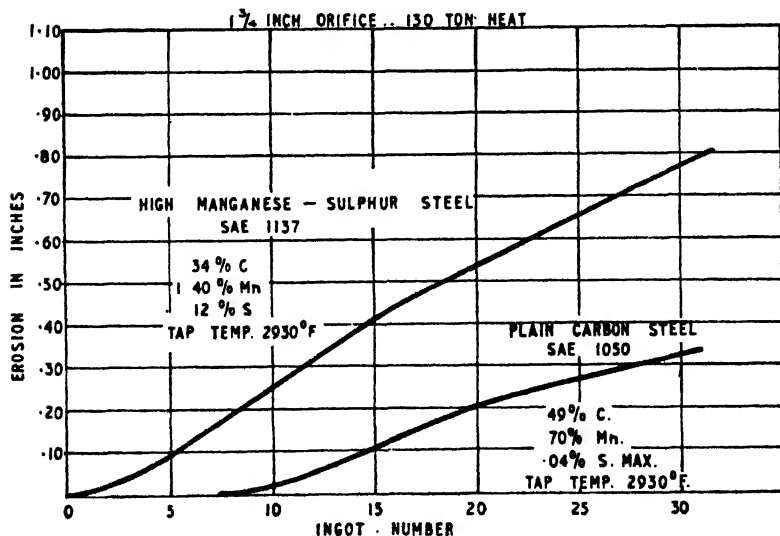


Fig. 255. Effect of steel composition on erosion of clay nozzles (after Ekholm and Hower).

be convenient if the rate of pour could be varied after the nozzle was opened, but the only method normally available, *viz.*, part closure, is far from satisfactory, since it tends to increase erosion. An alternative method recently developed in 1942 by Bethel and Bagnall, and applied to electric furnace steel, consists in having two or more nozzles, fitted one below the other, and so arranged that the smaller lower nozzle can be removed and pouring rate increased should this prove desirable at some stage in teeming.

## LADLE SLEEVES

### (a) CONSTRUCTION

The steel stopper rod is protected from the surrounding metal and slag by a series of fireclay tubes known as sleeve bricks or rod covers (*see* fig. 244, and fig. 256, p. 596). The external diameter of the sleeve varies from as little as  $3\frac{1}{2}$  in. to as much as  $6\frac{1}{2}$  in., and the corresponding internal diameter from  $1\frac{3}{4}$  in. to  $2\frac{1}{2}$  in. Both these extremes and four intermediate sizes have recently been the subject of a B.S.I. Specification (No. 2496: 1954). In this specification the use of a tapered spigot and socket are preferred, to the tongue and groove joint. The sleeve next to the stopper-end is frequently larger in diameter than the remainder, whilst a larger sleeve is also often used at the top of the rod to give additional protection in the slag zone. The space between the inside of the sleeve and the rod itself is sometimes filled with sand, whilst cement is normally employed between the individual sleeves.

Great care must be taken in assembly to ensure that no cracked sleeves are used and adequate expansion allowed. After the stopper-end has been fitted the sleeves are slid on over the rod and the joints made up one at a time, a gap being left between the top sleeve and the nut on the stopper rod to permit of expansion. Alternatively a spring washer may be employed. In many shops the stopper rods are dried by leaning them against the furnace but in some of the more modern installations specially designed ovens are used, the rods being left in the oven for 24 hours or more before use.

### (b) MATERIALS

There has been very little change in the properties of ladle sleeves during recent years, probably because these are in any case a compromise, high porosity, for example, being required to keep the rod cool but low porosity to resist slag attack. It will be seen from the properties given in Table LXIII that the refractoriness of British ladle sleeves is normally quite high and that difficulties of the type described by

TABLE LXIII  
PROPERTIES OF LADLE SLEEVES

	A	B	C	D	E
Apparent porosity, per cent.	29.0	22.0 (badly cored)	16.6	15.3	21.5
Bulk density, g.p.ml.	1.90	2.03	2.07	2.05	2.10
Apparent solid density, g.p.ml.	2.67	2.60	2.49	2.42	2.67
Permeability to air, c.g.s. units—through 2 skins	0.009	0.0003	0.00081	0.0017	0.0045
Permanent linear change on reheating, 2 hrs. @ 1410°C.	+3.3 3, 3	+2.7 1, 1	+2.3 5, 5	-0.2 5, 5	+2.3 11, 8
Thermal shock resistance (900°C. test) (2 in. section)	ave. 3 1675	ave. 1 1665	ave. 5 1720	ave. 5 1690	ave. 10 1680
Refractoriness, °C.	8	20	2	5	8
Free quartz (per cent.): estimated by microscope					
Chemical analysis (per cent.):					
SiO <sub>2</sub>	56.38	62.26	53.64	56.72	55.65
FeO	5.44	4.48	2.84	3.21	4.62
Al <sub>2</sub> O <sub>3</sub>	35.08	30.29	38.96	36.30	35.05
TiO <sub>2</sub>	1.16	1.03	1.50	1.29	1.29
CaO	0.22	0.12	0.04	—	0.06
MgO	0.83	0.72	1.60	1.32	1.50
Loss on ignition	0.12	0.28	—	—	—

Heindl and Cooke, which were associated with melting points as low as 1555°C. are, therefore, not experienced. It will be seen that the apparent porosities vary over a wide range—15.3 to 29.0 per cent. for the present samples.

Another reason for preferring higher porosity than would normally be found in, say, the stopper-end, is that ladle sleeves are peculiarly sensitive to thermal shock, particularly since they are liable to be laminated (*see* fig. 257, p. 605) during manufacture. No satisfactory method has yet been suggested of testing their thermal shock resistance, the data given in Table LXIII being obtained on a 2 in. transverse section sawn from the rod cover.

Differences in firing temperature can have a remarkable influence on the properties of rod covers, as is shown by the data given below:

	<i>Firing Temperature of Rod Covers</i>	
	1000°C.	1100°C.
Apparent porosity, per cent.	27.2	19.0
Bulk density, g.p.ml. . .	1.93	2.05
Permeability to air, c.g.s. units (2 skins) . . . .	0.0076	0.0030
Thermal shock resistance— 900°C. test . . . .	16 15 14	5)6 6)6
Mineralogical constitution (X-ray data) . . . .	{ Quartz, $\gamma$ alumina, Quartz, cristoba- small amount of lite, large amount mullite. of mullite.	

It so happens that with this particular clay even a small change in firing temperature results in a remarkable change in constitution, which is brought out most clearly by means of Debye Scherrer X-ray studies. Fig. 258, for example, shows the changing constitution when the firing temperature rises from 1000°C. to 1250°C. The soft-fired sample is seen to contain a good deal of free quartz, but on reaching 1250°C. this has virtually disappeared; instead there is a marked development of the mullite pattern and an increasing intensity in the cristobalite lines. Since most casting-pit refractories are fired in roughly this temperature range the use of X-rays can often provide extremely valuable information of firing treatment, particularly if adequate standards for the different clays and firing temperatures are available.

Where the conditions are such that even the largest rod covers are cut through by the slag, the desirability of using unfired basic refractories should be considered. Thus C. H. Bacon reports that at Messrs. John Summers, where unfired stabilised dolomite sleeves have been in use for many years, negligible trouble has been experienced with rod cover failure, whereas previously flying taps were quite common due

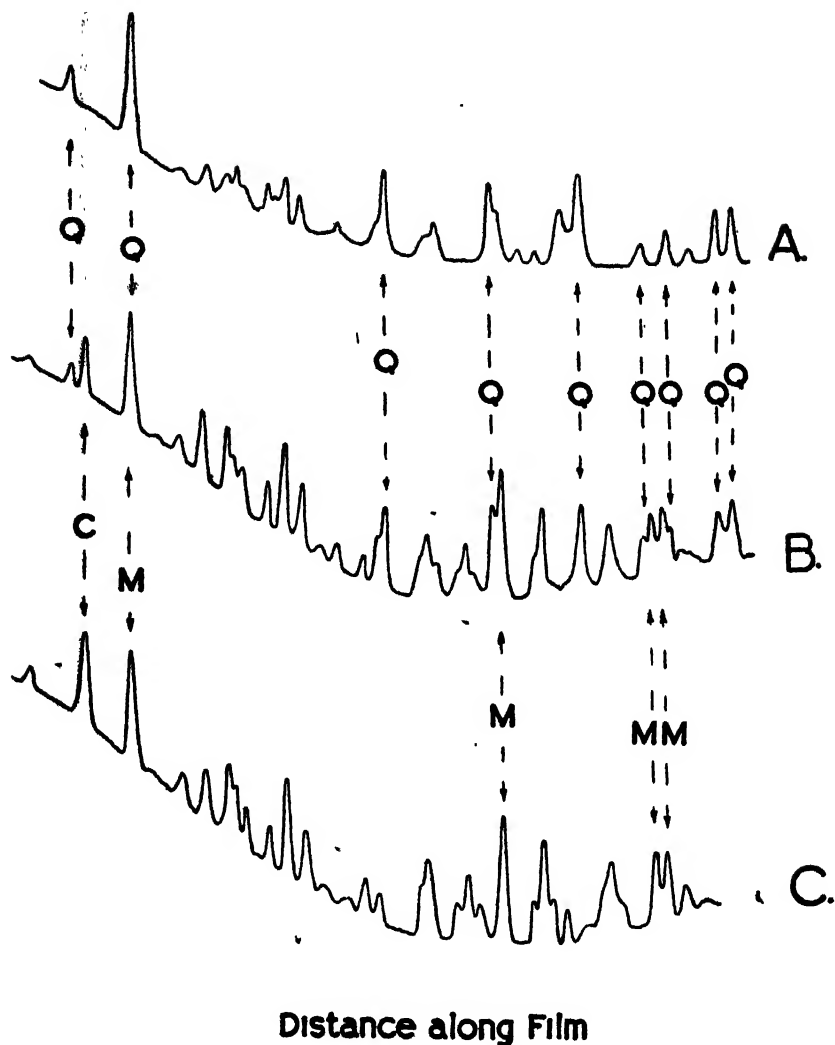


Fig. 258. Photometer curves of Debye-Scherrer X-ray photographs of siliceous fireclay casting pit refractory fired at: (A) 1000°-1050°C., (B) 1100-1150°C., and (C) 1250°C. The arrows indicate the prominent lines of quartz (Q), cristobalite (C), and mullite (M).

to the high iron oxide slag of low carbon steels cutting through the fireclay sleeve. Erosion with dolomite sleeves has been found to be negligible in the slag layer, whilst attack in the metal layer although appreciable is not as great as with fireclay. Some additional reaction

is found at the junction of fireclay sleeves with dolomite sleeves but this is not found to present any real problem provided the junction is kept just below the slag-metal interface.

(c) LIFE AND CAUSES OF FAILURE

Since a rod cover is in any case only used once—generally cracking badly on cooling down—the operator's sole concern is with its reliability. Certain operators fit twin stopper rods and nozzles but in general these are used to speed up teeming rather than as a safety measure. The slag resistance of a stopper rod is presumably governed by factors similar to those controlling the life of ladle bricks but freedom from spalling is more vital and tends to result in the use of more porous and more lightly fired material.

(d) LINES OF IMPROVEMENT

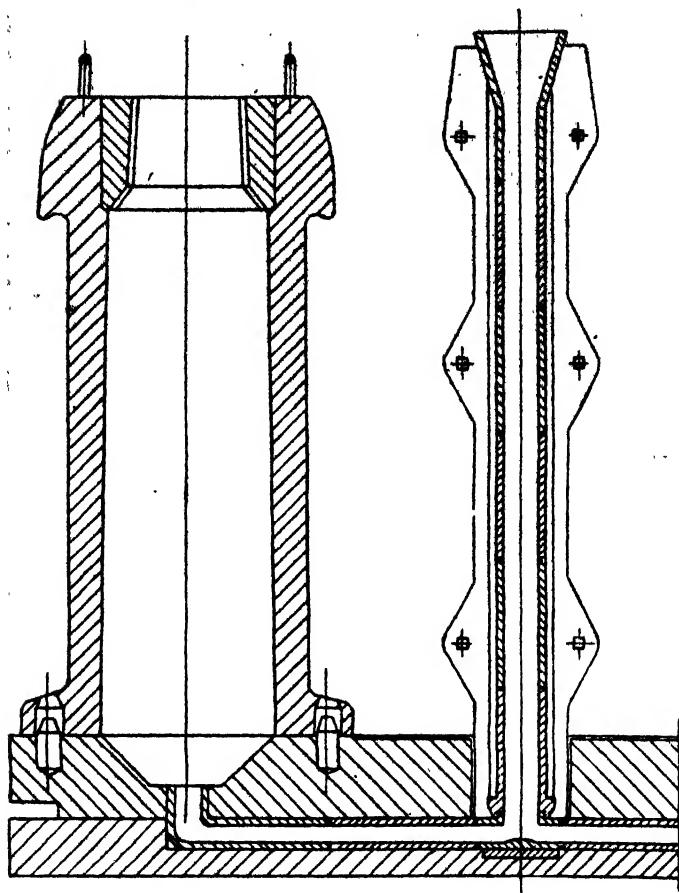
The combination of fireclay and stabilised dolomite rod covers described above would appear adequate for all normal conditions, though there is no doubt that the availability of far superior rod covers, nozzles and stoppers, might result in new applications, *e.g.*, better pouring control in the continuous casting process.

## TRUMPETS AND GUIDES

If the steel is direct poured there are no more refractory problems to be faced in the pit other than those of the mould top and occasionally a tundish. If on the other hand it is to be bottom poured then the refractory problems have just begun, since the steel must pass through a complex system of pipes (*see* fig. 259), starting with a trumpet-shaped fireclay funnel and passing *via* guide tubes, centre brick and various runner bricks, to the base of the mould. The centre brick (*see* fig. 260, p. 606) has a number of offtakes which are used to enable a number of moulds to be filled simultaneously.

The trumpet and guide bricks are held in position by a split cast-iron mould, similar in internal contour to the external contour of the guides and trumpet. The clearance between cast iron and refractory may be quite small, in which case the refractories are set dry, or may be quite substantial, in which case ganister cement must be used, at least at the joints. As might be expected, the internal diameter of the guides is considerably greater than that of the individual runners which it supplies. Thus the British Standard Specification No. 2496 shows an internal diameter varying from  $2\frac{7}{8}$  in. to  $2\frac{1}{4}$  in. compared with a maximum of 2 in. of runner bricks. The corresponding external diameters

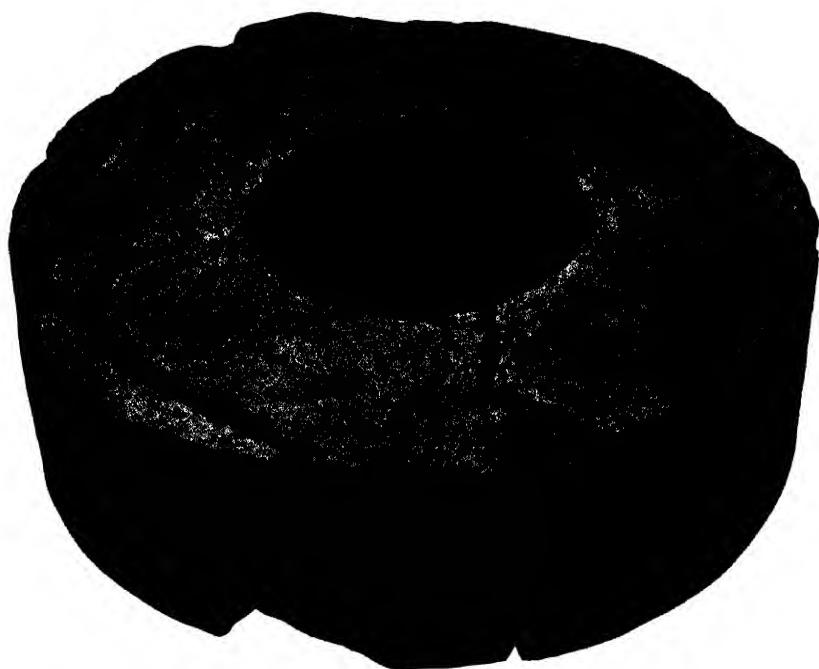




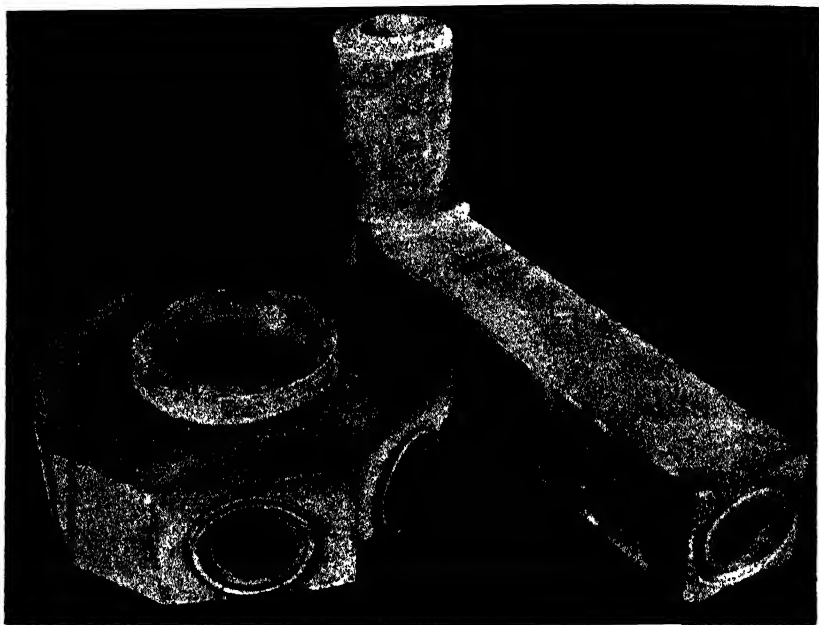
*Fig. 259. Arrangement of guide tubes, centre brick and runners for bottom pouring. One ingot mould only is shown.*

run from  $4\frac{1}{8}$  in. to 6 in., whilst the length of guides varies from 12 in. to  $15\frac{1}{2}$  in. Here again a spigot and socket type joint is preferred. Alignment of the trumpet and guides is so important that in some plants they are initially set up on a central rod which is only removed after the assembly has been correctly located in the split cast iron mould.

Table LXIV gives the properties of a number of guide tubes, a comparison of these with the corresponding data for runner bricks, as given in Table LXV, shows them to be essentially similar. A vital property, as given in the B.S.I. specification, is the accuracy of size and shape; any ovality or appreciable departure from specified dimensions greatly increasing the risk of a breakout. Bricks from one kiln will



*Fig. 257. Laminations in section through rod cover revealed by thermal shock test.*



*Fig. 260. Centre or crown brick with end runner.*



*Fig. 263. 32% alumina guide tube after use in casting manganese-molybdenum steel.*

TABLE LXIV

## PROPERTIES OF GUIDE TUBES

	A	B
Apparent porosity, per cent. . . . .	20.0	19.4
Bulk density, g.p.ml. . . . .	2.13	2.11
Apparent solid density, g.p.ml. . . . .	2.67	2.62
Permeability to air c.g.s. units.		
Perp. and through both skins . . . . .	0.0013	0.00022
Permanent linear change on reheating,		
2 hours @ 1410°C., per cent. . . . .	2.3 (bloated)	2.8 (bloated)
Thermal shock resistance, 2 in. section		
of 6 in. external diam. guides . . . . .	5	2
Refractoriness . . . . .	1670°C.	1660°C.
Free quartz (per cent.), estimated by		
microscope . . . . .	4	4

generally show essentially similar dimensions, though distortion may occur, particularly in the lower courses, but appreciable variation may be found between guides fired in different kilns and certainly between bricks made by different makers to the same drawing. Careful checking of supplies on arrival is, therefore, essential if breakouts are not to occur. Incidentally it should be assumed that all the guides will crack in service, though they should not show any signs of shattering when the steel passes through them. The actual wear on guide bricks is slight but they doubtless contribute their quota to the non-metallic inclusions entering the mould.

## RUNNERS AND CENTRE BRICK

When the steel leaves the guide tubes *via* the centre brick it usually divides into 2, 4, 6 or 8, streams, each of which is contained in a fireclay runner brick set in grooves in a cast iron plate. Here again accuracy of shape and size is important, though not quite as vital as with guide tubes. The runners are often set dry, the top face—and particularly the joints—are generally covered with cement prior to placing the cover plates and ingot moulds in position. Only the simplest types have been subject to specification (B.S.I. 2496), the internal diameters shown varying from 1½ in. to 2 in. and the lengths from 8 in. to 14 in. The external section is normally 3½ in. or 3½ in. square. Once again spigot and socket joints are specified.

TABLE LXV  
PROPERTIES OF RUNNER BRICKS

	A	B	C	D	E	F
Apparent porosity, per cent.	22.0	20.9	17.5	30.5	28.1	20.4
Bulk density, g.p.ml.	2.09	2.04	2.11	1.85	1.89	2.07
Apparent solid density, g.p.ml.	2.68	2.58	2.55	2.68	2.63	2.59
Permeability to air, c.g.s. units (2 skins) ..	0.00096	0.00074	0.0026	0.0012	0.015	0.0084
Permanent linear change on reheating, per cent. (2 hours @ 1410°C.) ..	+0.9	+2.7	+2.5	-1.1	+3.9	+2.0
Thermal shock resistance (900°C. test), (2 in. section) ..	4, 4	2, 7	4, 3	4, 5	10, 11	7, 12
Refractoriness ..	avge. 4	avge. 4	avge. 4	avge. 5	avge. 11	avge. 10
Free quartz (per cent.—estimated by microscope) ..	1690°C.	1700°C.	1730°C.	1690°C.	1700°C.	1720°C.
Chemical analysis (per cent.):	10	13	2	12	8	4
SiO <sub>2</sub> ..	56.84	57.10	53.46	58.41	55.79	56.26
FeO ..	5.12	3.36	2.82	2.95	3.21	2.57
Al <sub>2</sub> O <sub>3</sub> ..	(Fe <sub>2</sub> O <sub>3</sub> )					
TiO <sub>2</sub> ..	35.14	37.00	39.13	34.36	36.24	36.67
CaO ..	0.72	0.74	1.29	1.29	1.32	1.40
MgO ..	0.26	0.24	0.06	—	—	—
Loss on ignition ..	0.53	0.58	1.03	1.19	1.23	1.37
	0.16	0.22	—	—	—	—

The properties of a number of runner bricks are given in Table LXV, from which it will be seen that all the samples tested had a refractoriness of at least 1690°C. or over. The use of such refractory runners is, however, by no means universal, *e.g.*, Körber in Germany prefers a siliceous brick and claims that it is more resistant to attack. Just how many of the differences observed by numerous workers between low and high alumina runner bricks are in fact a function of alumina rather than physical texture is not yet demonstrated, but there is no doubt that the actual wear tends to be less with the more aluminous types provided they are made sufficiently dense, *e.g.*, by de-airing. Table LXV shows that in practice the bulk density and porosity may vary greatly from one supplier to the other, though the permeability tends to be always rather low. Thermal shock resistance is difficult to test, the only method at the moment being the 2 in. section and, as with guide tubes, freedom from cracking cannot be expected but shattering should certainly not occur.

Both our own observations and those of numerous other workers indicate that the attack by steel on runner bricks tends to be selective—in particular the manganese in the steel appears to go for the silica, forming MnO, which in turn combines with alumina and silica to form a low melting point glass. L. Halm gives for example the data shown in Table LXVI. Here the analyses of the reaction products are compared with the data of the unused brick. It would appear that some of the silica has been reduced by manganese in the metal (although this is only 0.4 per cent.) to silicon, with consequent increase in the alumina/silica ratio. As will be seen later, used runner bricks are sometimes found studded with crystals of corundum, but this probably comes from the aluminium added to the steel rather than from the refractories.

Centre bricks vary somewhat in design but the type shown in fig. 260, p. 606, is typical of the tongue and groove type assembly. Such bricks are hand made, the material used being similar to that employed for the runners.

## MOULD TOP BRICKS AND COMPO HEADS

Whether the steel is top or bottom poured the liquid surface tends to chill rapidly. This can be overcome in part by the use of anti-piping compounds (*see* Chapter VII) but mould top bricks can also be used to reduce the chill through the top of the mould side walls. A great variety of shapes and sizes are employed for this purpose some of which are shown in fig. 261. The simplest type consists of a refractory

TABLE LXVI

CHEMICAL ANALYSIS OF REACTION PRODUCTS BETWEEN REFRACTORY RUNNER AND STEEL

(after L. Halm)

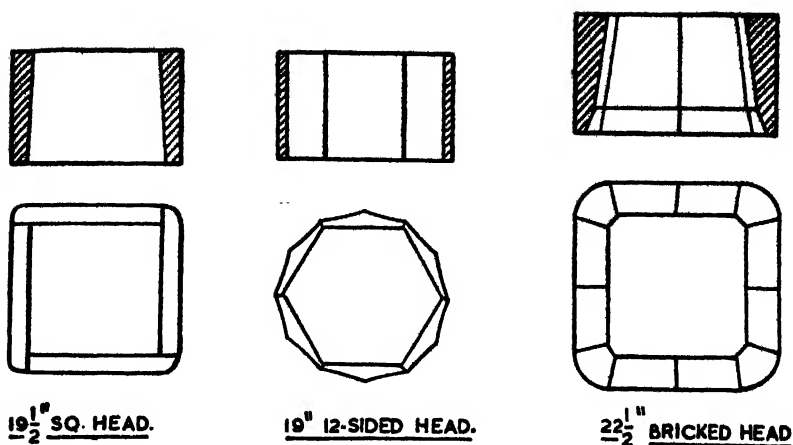
Type of steel poured: low carbon rimming steel

C = 0.07

Mn = 0.40

Si = 0.03

Chemical analysis (%)	Product			
	I	2	3	4
	Unused runner	Slag collected on ingot surface	Slag collected on the runner internal surface	Scorified zone of runner
SiO <sub>2</sub>	60.22	28.90	20.88	59.87
TiO <sub>2</sub>	1.36	35.65	45.45	{ 1.22
Al <sub>2</sub> O <sub>3</sub>	33.38	6.37	nil	{ 27.78
Fe <sub>2</sub> O <sub>3</sub>	2.54	10.44	5.64	2.49
FeO	—	17.83	26.00	1.18
MnO	—	0.73	0.78	4.98
CaO	0.79	0.15	nil	0.53
MgO	traces	0.66	n.d.	0.22
Na <sub>2</sub> O + K <sub>2</sub> O	1.43	2.08	1.34	1.94
Weight increase on ignition	—	0.85	0.47	—
Ratio SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	1.7			2.15



*Fig. 261. Selected designs of mould tiles and brick head.*

cylinder similar to a guide tube, but this is only employed with small ingots. In another arrangement four flat tiles with bevelled edges are fitted into a square mould and held apart at the corners by wooden wedges. These latter not only facilitate assembly, but act as expansion joints when hot metal rises in the mould and heats up the bricks. If bricks are fitted too tightly shattering may occur and large pieces fall into the steel and become trapped by the rising column of metal. Given close supervision and reasonably fluid steel such lumps will ultimately float, but examples have been seen where pieces of an inch or more across have been trapped half-way up the ingot. Mould top bricks, like rod covers, should have good spalling resistance and, therefore, demand good control during firing. Overheating, or "flashing" of the ware by flame contact may easily lead to low thermal shock resistance under service conditions. Spalling may also arise due to moulding faults which are very liable to occur in tiles due to the difficulties associated with their manufacture.

The properties of a number of mould top bricks are given in Table LXVII. It will be seen that they all happen to be of high refractoriness, though this is not an essential characteristic. What is important is that they should have a good thermal shock resistance and a fair insulating value, which in turn suggests a relatively high porosity.

Mould tops are by no means always lined with tiles, special heads being used that are either lined with bricks or with a ramming material, such as a grog-fireclay mix. Numerous attempts are being made to improve the insulating value of mould tops, *e.g.*, by waffle type tiles,



TABLE LXVII  
PROPERTIES OF MOULD TOP BRICKS

	A	B	C	D
Apparent porosity, per cent.	32.6	26.7	30.2	27.7
Bulk density, g.p.ml.	1.80	1.91	1.86	1.91
Apparent solid density, g.p.ml.	2.68	2.60	2.67	2.64
Permeability to air, c.g.s. units (through 2 skins)	0.018	0.012	0.0098	0.0048
Permanent linear change on reheating, per cent. (2 hours @ 1410°C.)	+4.2	+1.8	+2.1	+0.7
Thermal shock resistance: (900°C. test)	10, 9	30 +, 14	4, 5	10, 26
(3 in. × 2 in. × 1½ in. section)	ave. 10	ave. 22 +	ave. 5	ave. 18
Refractoriness	1680°C.	1680°C.	1680°C.	1710°C.
Free quartz (per cent.): estimated by microscope	10	8	18	8
Chemical analysis (per cent.): SiO <sub>2</sub>	57.46	59.10	62.54	54.52
FeO	4.88	5.04	4.40	3.44
Al <sub>2</sub> O <sub>3</sub>	34.89	32.80	30.10	39.28
TiO <sub>2</sub>	0.99	0.98	1.04	1.15
CaO	0.48	0.50	0.28	0.52
MgO	0.83	0.72	0.68	0.90
Loss on ignition	0.30	0.32	0.32	0.28

the use of insulating materials, or of combustibles which actually provide heat in a similar manner to anti-piping compounds.

### TUNDISH TILES

Where a particularly steady stream is required or more than one ingot is to be poured at once, single or multiple tundishes are employed. Types having 1 to 4 exits are shown in fig. 262. A wide variety

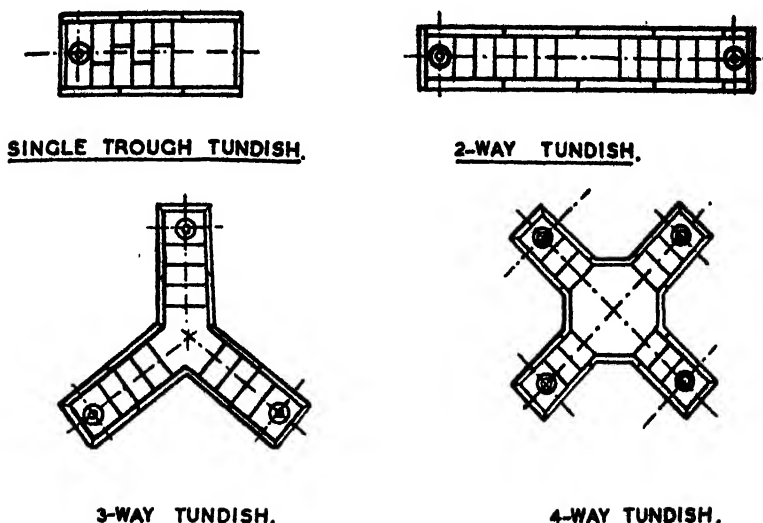


Fig. 262. Tundishes used to supply a steady stream of metal to one, two, three or four ingots.

of bricks would doubtless be satisfactory in this position, but good thermal shock resistance is certainly desirable and is shown by the two bricks tested (see Table LXVIII).

TABLE LXVIII  
PROPERTIES OF TUNDISH TILES

	A	B
Apparent porosity, per cent. .. ..	33.1	25.4
Bulk density, g.p.ml. .. ..	1.79	1.88
Apparent solid density, g.p.ml. .. ..	2.67	2.53
Refractoriness, °C. .. ..	1690	1670
Thermal shock resistance, 3 in. × 2 in. × 2 in. section .. ..	30+	30+

## CONTINUOUS CASTING

The experiments now in progress in continuous casting of steel will, if successful, eliminate many casting pit refractory problems completely, by avoiding the use of trumpets, guide tubes, centre bricks, runner bricks and mould tops, but will simultaneously introduce a new problem, *viz.*, the production of a nozzle capable of withstanding extreme conditions without any serious wear. The fact that zirconia and zircon in spite of their high price are among the most generally used materials for this purpose shows the importance attached to obtaining high refractoriness and a good resistance to liquid steel.

The conditions faced by the nozzle in continuous casting from a tundish are particularly severe, in that heat loss during pouring must be minimised and for this reason both the tundish and the nozzle are heated to high temperatures for a period prior to casting. The most vital consideration, however, is constancy of bore, since any wear on the nozzle inevitably means inclusions in the steel—the possibility of floating as in ordinary ingot practice being non-existent—whilst any build-up on the nozzle, such as may occur with certain steels, will gradually result in slowing up the exit stream and, consequent interference, if not actual stoppage, of the casting process itself.

Considerable work has already been done on the properties of super refractories for such nozzles, but the field is still so new that detailed reporting might well prove misleading.

## NON-METALLIC INCLUSIONS

During recent years particular attention has been paid to the cleanliness of special steels, such as are employed in aircraft construction. To the casual observer certain of the precautions, such as vacuum cleaning of bottom pouring settings, or allowing ladles to stand for given times before teeming, may appear a trifle fussy, but the vital character of the product necessitates every step being taken that might conceivably reduce the risk of premature failure. In spite of this position it is a fact that such steels are sometimes top poured and sometimes bottom poured and for certain qualities there does not appear to be any appreciable difference between the products, although in one case the steel passes through yards of additional refractory piping all of which can be observed to have been corroded by it. The moral from this and similar observations would seem to be that it is not so much what goes into the steel that matters as what stays in. One operator, convinced that refractories were frequently blamed for inclusions that had their origin elsewhere, demonstrated this point very vividly by pouring about 8 lb.

of crushed brick into the trumpet of a bottom pouring set-up. The ingots from this group were found on examination to be just as clean as the remainder.

Exceptions to the above generalisations must clearly be made for continuous casting, where everything that enters the steel remains in it, and to a less extent for bottom poured small ingots, where Rait and Pinder have pointed out that rapid freezing may well lead to a more effective trapping.

### *High versus low alumina casting-pit refractories*

From the manufacturers angle the principal issue is whether the use of first-class clays is justified for bottom pouring assemblies, since certain users get excellent results with the more siliceous materials. A carefully arranged trial was carried out by The United Steel Companies Limited in 1943 to get an answer to this question, one manufacturer agreeing to produce two assemblies differing merely in the clay from which they were made. The actual analyses of the two types, which are referred to as 32 and 40 per cent. respectively, were as follows:

				32% $Al_2O_3$	40% $Al_2O_3$
				%	%
$SiO_2$	..	..	..	58.50	53.00
$Fe_2O_3$	..	..	..	6.65	4.55
$Al_2O_3$	..	..	..	31.45	39.45
$TiO_2$	..	..	..	1.30	1.33
MnO	..	..	..	Nil	Nil
CaO	..	..	..	0.43	0.40
MgO	..	..	..	1.43	1.03

Physical tests made on the same bricks showed very similar bulk densities but an expected marked difference in refractoriness, the 32 per cent. alumina type melting at 1650°C. and the 40 per cent. alumina at 1720°C. The test cast, which was poured through the two assemblies simultaneously, was a manganese molybdenum steel made in a basic open-hearth furnace; its percentage analysis was: carbon 0.37, manganese 1.53, silicon 0.16, molybdenum 0.27. The rather heavy aluminium additions to this 80-ton cast were 51 lb. in the ladle and 19 lb. in the trumpets. The scums on the ingots were relatively slight and showed the following compositions:

				32% $Al_2O_3$ side	40% $Al_2O_3$ side
				%	%
$SiO_2$	..	..	..	28.5	26.5
FeO	..	..	..	6.5	19.3
$Al_2O_3$	..	..	..	41.0	33.8
$TiO_2$	..	..	..	0.7	1.0
MnO	..	..	..	20.9	13.5
CaO	..	..	..	0.5	not determined
MgO	..	..	..	0.9	„

They suggest that there has been substantial reaction between manganese in the metal with refractory, and also an appreciable pick-up of iron oxide. What is particularly interesting is the marked increase in the alumina/silica ratio. Cleanliness determinations made by the Fox Count showed no significant difference between the steels cast in the two assemblies, whilst mechanical tests, *e.g.*, yield point, maximum stress, and Izod, again showed negligible differences. Samples of slag chipped off the surface of the guide tubes (*see* fig. 263, p. 606) which are liable to be contaminated by some unused brick, showed the following percentage compositions:

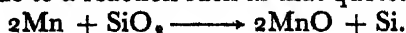
		32% $Al_2O_3$		40% $Al_2O_3$	
		1st Guide	5th Guide	1st Guide	5th Guide
		Down	Down	Down	Down
		%	%	%	%
$SiO_2$	..	34.5	29.5	18.6	29.4
FeO	..	6.9	7.6	11.9	14.1
$Al_2O_3$	..	38.5	41.8	49.5	35.0
$TiO_2$	..	0.9	1.0	0.8	0.7
MnO	..	17.0	17.0	17.6	18.9
CaO	..	0.6	0.5	0.7	N.D.
MgO	..	1.1	1.3	0.8	N.D.

Petrological examination of these refractories showed:

- a layer of well developed mullite next to the original brick,
- an intermediate zone of almost colourless glass,
- well developed crystals of corundum in the glass surface.

Here and there a few crystals of quartz were observed which had presumably been washed out of the refractory.

The above information suggested that attack on the refractories was not simply the result of attack by MnO and FeO in the metal but more likely to be due to a reaction such as that quoted by Körber:



The data given by Körber in fig. 264, for the manganese-silicon equilibrium for the varying carbon contents shows that the manganese/silicon ratio for this steel is above the equilibrium value, and oxidation of manganese by silica may, therefore, be expected. The greater amount of slag observed on the low alumina runners is presumably due to their higher content of silica. The presence of corundum crystals dotted over the surface of the glass is presumably due to the oxidation of metallic aluminium and the adherence of the product to the sticky surface of the runner. A study of the gits shows beyond doubt

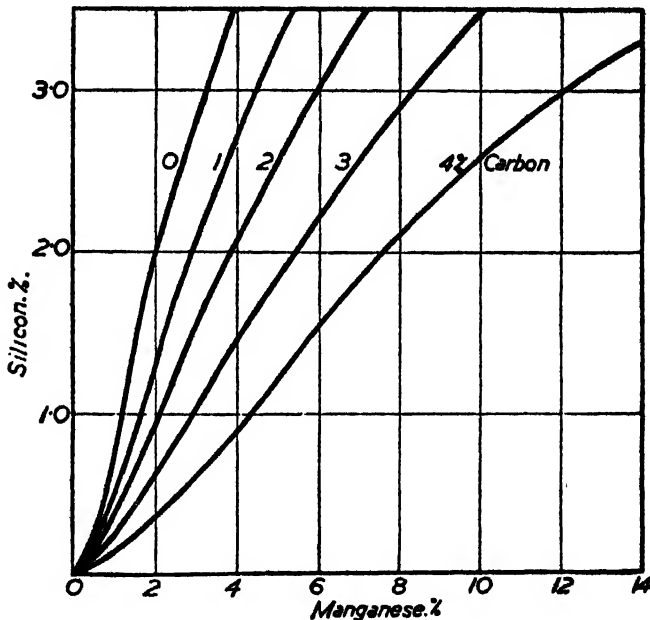


Fig. 264. Manganese-silicon equilibrium for varying carbon contents at an average temperature of 1600°C (after Körber).

that some of the manganese-alumina-silica glass formed by reaction between steel and refractory is carried into the mould, but there is little doubt that most of it rides to the top as scum, and that such conditions as metal superheat will determine cleanliness at least as much as refractory quality. That refractories are, however, responsible for at least part of the dirt in steel is shown by the presence of quartz in inclusions removed by the Dickenson method.

In their 1946 paper, Rait and Pinder have made a far more exhaustive study of the origin and constitution of non-metallic inclusions,

being particularly concerned with their effect on mechanical tests, and especially on their transverse strength. They divide the main sources into:

- (1) Deoxidation and the segregation of the products of deoxidation,
- (2) The presence of sulphur and phosphorus and the segregation of their compounds,
- (3) Extraneous sources.

In the first group they include alumina, arising from the use of aluminium as a final deoxidant, or for controlling grain size, and stress that its presence can be very troublesome in machining and drilling. In the third group they give inclusions arising from:

- (a) the trapping of slag,
- (b) the trapping of corroded or eroded refractories,
- (c) the trapping of reaction products between steel and refractories.

They made use of X-ray and microscopic examination, backed up by phase diagram data, to classify the non-metallic inclusions formed in alloy steel casts with uphill pouring and top pouring practice. They also report the "thick layer of dark green glassy slag" formed on the runner brick surfaces – particularly at the top – and again suggest that the attack of manganese on silica and particularly on quartz is responsible for it. Silica when present in the brick as mullite or as cristobalite they consider to be less reactive. In their trials, where relatively small ingots and, therefore, high cooling speeds were concerned, a marked reduction in the amount of manganese aluminosilicate inclusions was found when the more aluminous refractories were employed. Incidentally they state that during forging these inclusions are in a plastic state and generally are elongated into stringers parallel to the grain flow. They suggest that to reduce inclusions:

- (1) firebricks of 38 per cent. or more alumina content should be employed in the whole of the bottom pouring assembly,
- (2) fireclay nozzles should be boiled in tar, which they suggest decreases the manganese reduction of silica,
- (3) the use of aluminium be limited by adequate pre-oxidation of the steel by silicon,
- (4) research be done on basic ladle linings with a view to reducing inclusions of the gehlenite type,
- (5) large refractory inclusions be avoided by particular attention to the spalling resistance of refractories used in mould tops.

As far back as 1946 attempts were made by The United Steel Companies to use thoria as a radioactive tracer. For reasons still not completely understood bricks made with 5 per cent. additions showed very little activity after firing and the method was, therefore, abandoned in favour of a spectroscopic technique using 2 per cent. beryllium oxide as the tracer. Although these experiments were all of a very simple character they at least appeared to show that the 34 per cent.  $\text{Al}_2\text{O}_3$  refractories used were only responsible for a relatively small proportion of the inclusions in the particular cast examined.

Given an adequate tracer technique it should be possible to demonstrate not only what proportion of the inclusions come from the refractory but also which particular parts of the assembly, *e.g.* the ladle lining, nozzle or the runner bricks, are responsible. Fedock, for example has shown by using cobalt as a tracer and spectroscopic methods for identification, that the bulk of the inclusions arising from the ladle came from a region within about 20 in. of the nozzle centre. There is little doubt that such observations will be rapidly multiplied when radioactive tracers are adopted, as they would have been long since were it not for the experimental difficulties associated with the relatively short half-life of radioactive aluminium and silicon. What is already clear is that the user will increasingly demand steel free from deleterious inclusions, and that in providing this the steelmaker will demand increasing assistance from his colleagues in the firebrick plants and possibly also from those concerned with the production of basic refractories.





# APPENDICES

## APPENDIX I

### GLOSSARY OF TERMS COMMONLY USED IN CONNECTION WITH REFRACTORIES AND FURNACES

- ABRASION:** The wearing away of a surface due to a rubbing action, *e.g.*, by dust-laden gases, or slabs on a hearth.
- ABSORPTION:** The ratio of the weight of fluid (normally water) which can be absorbed by a material to the dry weight. Usually expressed as a percentage.
- ABUTMENT:** The structural member which withstands the thrust of an arch. This generally consists of a skewback brick and a steel support.
- AFTER-CONTRACTION OR EXPANSION:** See Permanent Linear Change on Reheating.
- AIR-SETTING CEMENT:** A cement developing a strong bond on drying but without heat.
- AMORPHOUS:** Having no crystalline structure.
- ANISOTROPIC:** Having a crystal structure other than cubic; giving rise to more than one index of refraction.
- ARCH:** A furnace part supported at its ends only and formed of a number of tapered bricks held in place by the keying effect of the taper.
- ARCH-FLAT:** An arch in which both the outer and inner surfaces are horizontal.
- ARCH-SPRUNG:** An arch only supported by abutments whose cross-section usually consists of a segment of a circular ring.
- ARCH-SUSPENDED:** An arch built from bricks hung from overhead supporting members.
- BATTER:** The slope of the face of a wall; also applied to the angle at which the face of a wall slopes from the vertical.
- BIREFRINGENCE:** The property of an anisotropic crystal viewed under crossed nicols whereby certain characteristic colours are produced which are a measure of the difference between the maximum and minimum values of the indices of refraction of the crystal.
- BLACK BODY:** A body which at a given temperature absorbs all radiation falling on it. Such a body has an emissivity of 1.0. A closed furnace, the walls of which are at uniform temperature and the interior of which is viewed through a comparatively small hole, is such a black body.
- BLACK-HEART:** A defect sometimes produced in fireclay bricks due to vitrification of the surface before the organic matter in the interior has been oxidised. Black-hearted bricks may show a low slag resistance.
- BOND:** A material added to (or already present in) a refractory batch, whose function is to promote strength either in the green, dry or fired state. The same term is applied to the various methods of building bricks whereby adjacent courses are tied into one another.

- B.Th.U. (BRITISH THERMAL UNIT):** The amount of heat required to raise the temperature of one pound of water at its maximum density one degree Fahrenheit. It is equal to 252 calories.
- BUCKSTAY:** A steel member used in furnace construction to take the thrust of the refractory structure, *e.g.*, of a roof.
- BULK DENSITY:** The ratio of the weight of a material to its total volume (*i.e.*, including pore space). Expressed as g.p.ml. (g.p.c.c.) or lb. per cu. ft. (*see* Chapter I).
- BULKHEAD:** A panel of brick built into a wall for easy replacement.
- BUNG:** A removable roof section built in a metal frame.
- CALCINATION:** Heat treatment applied to certain rocks and minerals to effect dissociation (and/or produce a change in physical structure), *e.g.*, clay is calcined to drive off combined water.
- CALORIE (SMALL):** The amount of heat required to raise the temperature of one gram of water one degree centigrade from 3.5°C. to 4.5°C.
- CALORIE (LARGE):** The amount of heat required to raise the temperature of one kilogram of water one degree centigrade from 3.5°C. to 4.5°C.
- CAMPAIGN:** The working life of a furnace between major repairs.
- CASTABLE REFRACTORY:** A hydraulic setting refractory suitable for casting into shapes and usually bonded with aluminous cement.
- CATENARY ARCH:** An arch designed in the form of an inverted catenary, the arch thereby having a minimum of stresses. A catenary is roughly the curve taken by a thread suspended at each end.
- CERAMIC:** A general term applied to all materials made from clayey and earthy substances by the application of heat.
- CERMETS:** Mixtures of ceramic materials with metals. Also known as Cermals.
- COLLOIDS:** Very small particles of substances which remain in suspension in liquids. Certain clays are partly colloidal. True colloids are substances like starch and gelatin.
- COMPONENTS:** The number of components of a system is the smallest number of independently variable constituents by means of which the composition of each phase can be quantitatively expressed (*see also* PHASE, PHASE RULE).
- CONDUCTIVITY:** *See* THERMAL CONDUCTIVITY.
- CONGRUENT MELTING:** Applied to a solid which melts to give a liquid of identical composition.
- CORBELLING:** A method of building brick walls whereby each course of brickwork is displaced with respect to the course immediately below it.
- CORING.** *See* BLACK-HEART.
- CORROSION:** Wearing away of a material, *e.g.*, furnace brickwork, by chemical action of fluxes.
- CRYPTO-CRYSTALLINE:** A crystalline structure in which the crystals are too small to be readily studied under the microscope.

- CRYSTALLINE:** Applied to solids which have a definite and regularly repeated internal molecular structure.
- DEAD-BURNED:** Applied to materials which have been fired to a temperature sufficiently high to render them relatively resistant to moisture and free from excessive after-contraction, *e.g.*, dead-burned magnesite.
- DEAIRED:** The term applied to a brick which has been moulded or formed from a batch subjected to a partial vacuum.
- DEGREE OF FREEDOM:** The number of variables (temperature, pressure, concentration) which must be arbitrarily fixed to define a system completely (*see also* PHASE, PHASE RULE).
- DENSITY:** The weight of unit volume of a substance (*see also* BULK DENSITY).
- DEVITRIFICATION:** The formation of crystals in a material originally in the glassy state.
- DIFFUSIVITY:** The diffusivity of a material determines the rate at which the outside of a structure heats up when heat is applied to the inside. It is given by the formula  $\frac{k}{\rho C}$   
 where  $k$  = thermal conductivity  
 $\rho$  = density  
 $C$  = specific heat of brickwork.
- DISSOCIATION:** The breakdown of a compound usually under the influence of heat.
- DOUBLE BURNED:** A term misleadingly applied in the United States to brick or dolomite that has been once fired at a high temperature.
- EMISSION:** A measure of the energy radiated from unit area of a surface in unit time for unit difference of temperature between the surface in question and surrounding bodies, expressed as a fraction of the maximum possible radiation.
- EQUILIBRIUM:** Equilibrium exists in any system under a fixed set of conditions when the parts of the system do not undergo any change of properties with the passage of time, and provided the parts of the system have the same properties when the same conditions are again arrived at by a different procedure.
- EROSION:** The wearing away of a material. Usually applied to wear caused by physical rather than chemical forces; *cf.* CORROSION and ABRASION.
- EUTECTIC:** The composition having the lowest melting point in a series of two or more components.
- EXPANSION:** *See* THERMAL EXPANSION.
- FANTAIL:** The large flue joining the slag pocket to the regenerator on an open-hearth furnace.
- FLINT FIRECLAY:** A fireclay having a flint-like appearance and a very low plasticity.
- FLUX:** A material which lowers the fusion point of a refractory material.
- FUSION:** The softening of a solid material by heat alone or by the combined action of heat and fluxes.

- GANGUE:** Accessory minerals associated with relatively valuable minerals.
- GAP-SIZED GRADING:** A material from which the intermediate material has been screened, generally with the object of producing a brick of high bulk density.
- GREEN STRENGTH:** The strength of a ceramic body in the moulded but unfired state.
- GROG:** Non-plastic material, usually prefired, added to a brick batch to reduce drying and firing shrinkage, or obtain special properties, *e.g.*, high thermal shock resistance.
- GROUT:** A mortar-water suspension sufficiently thin to permit of flowing into vertical open joints on horizontal brickwork courses.
- HEADER:** A brick laid on the flat or on edge with its length perpendicular to the plane of a wall.
- INCONGRUENT MELTING:** Dissociation of a solid on heating to form another compound and liquid of a different composition from that of the original solid.
- INDEX OF REFRACTION:** A property of minerals useful in their identification. It is defined as the ratio of the velocity of light in the mineral to the velocity of light in air, and is usually determined by immersing particles of the mineral in liquids of known refractive index.
- INTERMEDIATE DUTY FIRECLAY BRICK:** An American classification for a fireclay brick having a pyrometric cone equivalent not lower than No. 29, or a refractoriness-under-load such that less than 3 per cent. deformation occurs in the A.S.T.M. test at 2460°F.
- INVERSION:** The physical change in structure between two or more forms of polymorphic crystals, *e.g.*, the inversion of  $\alpha$  quartz to  $\beta$  quartz at 575°C.
- ISOMETRIC:** Applied to minerals which crystallise in the cubic system, *i.e.*, have three equal axes at right angles and have identical properties with respect to these axes.
- ISOMORPHOUS:** Minerals of analogous chemical composition and similar crystal structure. Such minerals normally form solid solutions, *e.g.*,  $2\text{MgO} \cdot \text{SiO}_2$  and  $2\text{FeO} \cdot \text{SiO}_2$ .
- ISOTROPIC:** Having equal properties in all directions, *e.g.*, only one refractive index.
- JAMB:** A vertical structural member forming the side of an opening in a furnace wall.
- KEY BRICK:** A tapered brick used for closing and also tightening up a curved arch (*see* Appendix 17).
- KNUCKLE:** The junction of the port and main roof in an open-hearth furnace, which normally juts downwards.
- LIQUIDUS TEMPERATURE:** The temperature at which crystallisation commences when a fully melted material is slowly cooled, or melting is completed on heating up.
- LOW DUTY FIRECLAY BRICK:** One having a pyrometric cone equivalent not lower than cone 19.

**MICRON:** One-thousandth mm.

**MILLSCALE:** The black magnetic form of iron oxide (mainly  $\text{Fe}_3\text{O}_4$ ) formed on iron and steel when heated prior to rolling or forging.

**MINERAL:** "A mineral species is a natural inorganic substance which is either definite in chemical composition and physical characters or varies in these respects between definite natural limits."—Winchell.

**MINERALISER:** A small quantity of flux added to a brick batch or refractory aggregate to promote crystal growth or compound formation, *e.g.*, the lime added to silica brick batch to accelerate the conversion of quartz to tridymite and cristobalite.

**MODULUS OF RUPTURE:** The transverse strength of a material. It is given by the formula  $M = \frac{3wl}{2bd^2}$

where M = Modulus of rupture in lb. per sq. in.

w = Total load in lb. at which failure occurs.

l = Distance between supports in inches.

b = Width of test piece in inches.

d = Thickness of test piece in inches.

**MONKEY WALL:** The term applied to the section between the back and front walls of an open-hearth furnace and the port side walls, particularly when these protrude into the furnace.

**MONOLITHIC LINING:** A lining containing no joints which is formed by ramming or sintering into position a granular material.

**ORTON CONES:** Standard pyrometric cones as used in U.S.A. (*see* Appendix 6a).

**PACKING DENSITY:** The density of an aggregate packed in a container under controlled conditions (g.p.ml. or lb. per cu. ft.), *e.g.*, the packing density of moulding sands as determined with an A.F.A. rammer.

**PALLETISING:** A method of transporting bricks, in which the latter are stacked on wooden or metal boards to facilitate handling.

**PERMANENT LINEAR CHANGE ON REHEATING:** The linear contraction or expansion measured on a material reheated to a given temperature for a given time. Expressed as a percentage of the original length (*see also* AFTER-CONTRACTION OR EXPANSION).

**PERMEABILITY:** A measure of the rate at which a gas or liquid will pass through a porous body. Sealed pores take no part in permeability (*see* Chapter I).

**PETROLOGY:** The science of rocks.

**pH:** A measure of the acidity of a solution. Defined as the logarithm of the reciprocal of the hydrogen ion concentration. The pH of pure water is 7.0. Values less than 7.0 indicate acid and values greater than 7.0 alkaline solutions.

**PHASE:** A physically homogeneous but mechanically separable portion of a system.

**PHASE RULE:** In its simple form the phase rule can be expressed by:

$$F + P = C + 2$$

where C = number of components of system

P = number of phases present at equilibrium

F = degrees of freedom or variance of system

**PIG IRON:** Relatively impure cast iron produced by a blast-furnace.

**PIPE:** The cavity formed in metal due to contraction during solidification.

**PLASTICITY:** The property of a material by virtue of which it can be moulded into any desired form, and which retains that form when the pressure of moulding has been removed.

**PLUMBAGO:** Clay-graphite refractories.

**POINTING:** The insertion of mortar into unfilled joints after the bricks have been laid.

**POLYMORPHISM:** The property by virtue of which certain chemically identical materials form two or more minerals of different crystal structure and physical properties, *e.g.*, silica crystallises as quartz, cristobalite or tridymite.

**POROSITY:** The ratio of the volume of pores in a refractory body to the volume of the entire body. Usually expressed as a percentage (*see* Chapter I). If P = Porosity (per cent.), B.D. = Bulk Density (g.p.ml.) and A.S.G. = Apparent Specific Gravity, then

$$\text{Porosity} = P = 100 \left\{ 1 - \frac{\text{B.D.}}{\text{A.S.G.}} \right\}$$

**PORT:** An opening in a furnace through which fuel or air enter or exhaust gases escape.

**POWDER DENSITY:** The density of a material in powder form. This approaches the specific gravity and differs from the bulk density, which includes all the pores.

**PUG MILL:** The machine used for mixing clay or other material with water, usually consisting of a central shaft to which blades are fitted and which revolves in a trough.

**PYROMETRIC CONES:** Small pyramid-shaped pieces of mixtures of minerals which melt at definite temperatures under standardised conditions. They are used as a basis for comparison in the determination of the Pyrometric Cone Equivalent or refractoriness of refractory materials (*see* Chapter I, and Appendix 6a).

**PYROMETRIC CONE EQUIVALENT (P.C.E.):** In the determination of refractoriness, the test cone is heated up in company with standard cones whose deformation temperature is known. The P.C.E. value is that of the cone or cones which deform at the nearest temperature to that at which the test cone deforms. A cone is said to have deformed (or melted) when it has bent over until the tip is on a level with the base.

**PYROPLASTICITY:** High temperature plasticity due to softening or melt formation.

**RECUPERATOR.** A continuous heat exchanger in which heat is extracted from the products of combustion and returned to incoming air through metal or refractory walls.

- REDUCING ATMOSPHERE:** One having a deficiency of oxygen, such as occurs for example in parts of furnaces where combustion is incomplete and iron exists either as the metal or as ferrous oxide.
- REFRACTORINESS:** A term used as an index of the heat-resisting properties of refractories. It is usually determined on a sample in the form of a cone cut or prepared from the ground refractory (*see* PYROMETRIC CONE EQUIVALENT and Chapter I.)
- REFRACTORINESS-UNDER-LOAD:** A measure of the resistance of a refractory to the combined effects of heat and loading (*see* Chapter I). Often expressed as the temperature of shear or 10 per cent. deformation when heated up under 25 or 50 lb. per sq. in.
- REGENERATOR:** A cyclic heat exchanger which alternately receives heat from combustion products and transfers heat to air or gas used in combustion.
- RELIEVING ARCH:** A sprung arch provided in a wall above an opening to support the wall and reduce the strain on a second arch built below it.
- REPPRESSED BRICK:** A brick formed by repressing blanks cut from a stiff column of clay produced by an extruding machine.
- REVERBERATORY FURNACE:** One in which fuel is burned at one end and the gases pass over the charge before leaving by the stack.
- RHEOLOGY:** That branch of science which deals with the deformation and flow of matter.
- RISE:** The vertical distance between a plane connecting the spring lines and the highest point on the undersurface of an arch.
- SECONDARY EXPANSION:** The permanent expansion shown by certain fireclay bricks within their useful range of service.
- SEGER CONES:** Standard pyrometric cones as used in Great Britain and Germany (*see* Appendix 6a.)
- SILL:** Horizontal structural member forming the bottom of a door in a furnace wall.
- SKEWBACK:** A course of brickwork having an inclined face from which an arch is sprung.
- SKULL:** The crust of solid metal left in a ladle due to premature cooling of the metal.
- SLAG:** Material formed by fusion of oxides in metallurgical processes. May also be applied to fused reaction product between a refractory and a flux.
- SLIP CASTING:** Process in which the material to be cast is ground, mixed with sufficient water to give a creamy liquid, and then poured into plaster moulds which rapidly absorb the added water, leaving a solid body having the inside shape of the mould.
- SOLDIER COURSE:** A course of bricks laid on end.
- SOLID SOLUTIONS:** Certain groups of crystalline minerals have the property of dissolving in one another either in all proportions or over a limited range of composition. Such groups are said to form solid solutions.



**SPALLING:** "Breaking or cracking of refractory brick in service, to such an extent that pieces are separated or fall away, leaving new surfaces of the brick exposed."—A.S.T.M. definition.

**SPECIFIC GRAVITY:** The ratio of the weight of unit volume of a substance to the weight of unit volume of water.

**SPECIFIC HEAT:** The ratio between the amounts of heat required to raise the temperature of unit weight of a substance and of unit weight of water one degree.

**SPRINGER:** *See* SKEWBACK.

**STATISTICAL TERMS:** *See* Chapter I.

**STRETCHER:** A brick laid on the flat with its length parallel to the plane of the wall.

**SUPER DUTY FIRECLAY BRICK:** A fireclay refractory having a pyrometric cone equivalent not lower than No. 33, not more than 1 per cent. linear shrinkage in the 2910°F. reheating test, and not more than 4 per cent. loss in the panel spalling test (preheated at 3000°F. A.S.T.M. c.27-41).

**SUPER DUTY SILICA BRICK:** A term applied in the United States to a silica brick in which the total of alumina, titania and alkalis, is significantly lower than normal.

**THERMAL CONDUCTIVITY:** The property by virtue of which heat is transmitted through matter (*see* Chapter VII).

**THERMAL EXPANSION:** The increase in dimensions of a material when heated. The term is only applied to that part of the expansion which is reversible and should not be confused with the permanent expansion which occurs when some substances are heated (*cf* after-expansion).

**THERMAL SHOCK RESISTANCE:** The ability to withstand sudden heating or cooling without cracking (*see* Chapter I).

**THIXOTROPY:** The rate of change of viscosity of a suspension or slip as the rate of shearing strain varies. It has the units of intensity of stress.

**VITRIFICATION:** A process of conversion of a substantial part of a refractory body into a glass. It is normally accompanied by a reduction in porosity and an increased spalling tendency.

**WARPAGE:** The deviation from the intended surface of a refractory shape resulting from distortion during manufacture.

**WICKET-TEMPORARY:** A door built in a furnace or kiln, *e.g.*, the temporary closures on checker chamber ends.

**WIRE-CUT BRICK:** Brick cut by wire from a column of extruded clay and not repressed.

**WORKABILITY:** The combination of properties which enables refractory mortars, plastic refractories, or ramming mixtures to be used with a minimum of effort.

APPENDIX 2  
INTERNATIONAL ATOMIC WEIGHTS (1953)

<i>Name</i>	<i>Symbol</i>	<i>Atomic No.</i>	<i>Atomic Weight*</i>
Actinium .. .. .	Ac	89	227
Aluminium .. .. .	Al	13	26.98
Americium .. .. .	Am	95	(243)
Antimony .. .. .	Sb	51	121.76
Argon .. .. .	A	18	39.944
Arsenic .. .. .	As	33	74.91
Astatine .. .. .	At	85	(210)
Barium .. .. .	Ba	56	137.36
Berkelium .. .. .	Bk	97	(245)
Beryllium .. .. .	Be	4	9.013
Bismuth .. .. .	Bi	83	209.00
Boron .. .. .	B	5	10.82
Bromine .. .. .	Br	35	79.916
Cadmium .. .. .	Cd	48	112.41
Calcium .. .. .	Ca	20	40.08
Californium .. .. .	Cf	98	(248)
Carbon .. .. .	C	6	12.011
Cerium .. .. .	Ce	58	140.13
Cesium .. .. .	Cs	55	132.91
Chlorine .. .. .	Cl	17	35.457
Chromium .. .. .	Cr	24	52.01
Cobalt .. .. .	Co	27	58.94
Columbium ( <i>see</i> Niobium) .. .. .			
Copper .. .. .	Cu	29	63.54
Curium .. .. .	Cm	96	(245)
Dysprosium .. .. .	Dy	66	162.46
Erbium .. .. .	Er	68	167.2
Europium .. .. .	Eu	63	152.0
Fluorine .. .. .	F	9	19.00
Francium .. .. .	Fr	87	(223)
Gadolinium .. .. .	Gd	64	156.9
Gallium .. .. .	Ga	31	69.72
Germanium .. .. .	Ge	32	72.60
Gold .. .. .	Au	79	197.0
Hafnium .. .. .	Hf	72	178.6
Helium .. .. .	He	2	4.003
Holmium .. .. .	Ho	67	164.94
Hydrogen .. .. .	H	1	1.0080
Indium .. .. .	In	49	114.76
Iodine .. .. .	I	53	126.91

\*A value given in brackets denotes the mass number of the isotope of longest known half-life.

APPENDIX 2  
INTERNATIONAL ATOMIC WEIGHTS (1953)—*continued*

<i>Name</i>	<i>Symbol</i>	<i>Atomic No.</i>	<i>Atomic Weight*</i>
Iridium .. .. .	Ir	77	192.2
Iron .. .. .	Fe	26	55.85
Krypton .. .. .	Kr	36	83.80
Lanthanum .. .. .	La	57	138.92
Lead .. .. .	Pb	82	207.21
Lithium .. .. .	Li	3	6.940
Lutetium .. .. .	Lu	71	174.99
Magnesium .. .. .	Mg	12	24.32
Manganese .. .. .	Mn	25	54.94
Mercury .. .. .	Hg	80	200.61
Molybdenum .. .. .	Mo	42	95.95
Neodymium .. .. .	Nd	60	144.27
Neptunium .. .. .	Np	93	(237)
Neon .. .. .	Ne	10	20.183
Nickel .. .. .	Ni	28	58.69
Niobium (Columbium) .. .. .	Nb	41	92.91
Nitrogen .. .. .	N	7	14.008
Osmium .. .. .	Os	76	190.2
Oxygen .. .. .	O	8	16
Palladium .. .. .	Pd	46	106.7
Phosphorus .. .. .	P	15	30.975
Platinum .. .. .	Pt	78	195.23
Plutonium .. .. .	Pu	94	(242)
Polonium .. .. .	Po	84	210
Potassium .. .. .	K	19	39.100
Praseodymium .. .. .	Pr	59	140.92
Promethium .. .. .	Pm	61	(145)
Protactinium .. .. .	Pa	91	231
Radium .. .. .	Ra	88	226.05
Radon .. .. .	Rn	86	222
Rhenium .. .. .	Re	75	186.31
Rhodium .. .. .	Rh	45	102.91
Rubidium .. .. .	Rb	37	85.48
Ruthenium .. .. .	Ru	44	101.1
Samarium .. .. .	Sm	62	150.43
Scandium .. .. .	Sc	21	44.96
Selenium .. .. .	Se	34	78.96
Silicon .. .. .	Si	14	28.09

\*A value given in brackets denotes the mass number of the isotope of longest known half-life.

APPENDIX 2  
INTERNATIONAL ATOMIC WEIGHTS (1953)—continued

Name					Symbol	Atomic No.	Atomic Weight*
Silver	..	..	..	..	Ag	47	107.880
Sodium	..	..	..	..	Na	11	22.991
Strontium	..	..	..	..	Sr	38	87.63
Sulfur	..	..	..	..	S	16	32.066†
Tantalum	..	..	..	..	Ta	73	180.95
Technetium	..	..	..	..	Tc	43	(99)
Tellurium	..	..	..	..	Te	52	127.61
Terbium	..	..	..	..	Tb	65	158.93
Thallium	..	..	..	..	Tl	81	204.39
Thorium	..	..	..	..	Th	90	232.05
Thulium	..	..	..	..	Tm	69	168.94
Tin	..	..	..	..	Sn	50	118.70
Titanium	..	..	..	..	Ti	22	47.90
Tungsten	..	..	..	..	W	74	183.92
Uranium	..	..	..	..	U	92	238.07
Vanadium	..	..	..	..	V	23	50.95
Xenon	..	..	..	..	Xe	54	131.3
Ytterbium	..	..	..	..	Yb	70	173.04
Yttrium	..	..	..	..	Y	39	88.92
Zinc	..	..	..	..	Zn	30	65.38
Zirconium	..	..	..	..	Zr	40	91.22

\*A value given in brackets denotes the mass number of the isotope of longest known half-life.

†Because of natural variations in the relative abundance of the isotopes of sulfur the atomic weight of this element has a range of  $\pm 0.003$ .



## APPENDIX 4a

## THERMOCOUPLES AND PYROMETERS

The main types of high temperature measuring devices used commercially are as follows:

- A. Thermocouples
- B. Total radiation pyrometers
- C. Optical pyrometers (disappearing filament and polarising types)
- D. Photo-electric cells
- E. Pyrometric cones
- F. Temperature indicating paints and crayons.

## A. THERMOCOUPLES

The main types used in the steelplant are:

1. Base metal couples—which are used up to about 1100°C. The most common type is the chromel-alumel couple. At low temperatures it has the advantage over a rare metal couple of high E.M.F.—temperature values.
2. Rare metal couples—which consist usually of platinum and platinum-rhodium alloy, and can be used up to 1600°C. The 13 per cent. Rh alloy is more generally favoured although the 10 per cent. Rh alloy is used to some extent in the U.S.A. The E.M.F. temperature values for U.S. and British couples using the 13 per cent. Rh alloy differ somewhat (*see* Appendix 4b). Special thermocouples of higher Rh content are now available and can be used up to 1850°C.

In using thermocouples it is important to protect them from mechanical shock. They are also susceptible to deterioration in certain gases and must be adequately protected, *e.g.*, with silica or porcelain sheaths. Rare metal couples are particularly susceptible to carbonaceous gases, especially when in contact with a silica-bearing refractory.

Thermocouples are conveniently calibrated by using a substance of standard melting point (*see* Appendix 5). They can also be checked against a standard couple.

## B AND C. PYROMETERS DEPENDING ON RADIATION EMITTED FROM A HOT BODY

The main advantage of these types of pyrometer is that they can be used at a distance from the body whose temperature is being measured. Correct readings are only obtained under black body conditions, but this does not limit their applications for many processes where *control* of temperature rather than absolute values is necessary. Corrections can be made if the conditions vary much from black body conditions. There are three main types of pyrometer in this class: the total radiation, the disappearing filament and the polarising type.

1. *Total radiation fixed or variable (focus) type.* This uses all the radiation falling on it, and has the great advantage that it can be coupled with a recorder. Such an instrument can be permanently fixed at a distance from a furnace. The main precautions to be taken in its use are to keep the lenses clean and the instrument cool. The calibration is usually

supplied by the manufacturers, but can be checked against a black-body furnace with a thermocouple in it. At higher temperatures it can be checked by applying the Stefan-Boltzmann radiation law, having determined the constant at a lower temperature.

2. *Optical pyrometers.* The two main types of optical pyrometers are the disappearing filament type and the less used polarising or Wanner type. Both can be used from red heat up to about 2000°C. They are useful for spot checking a recording instrument and for measuring temperatures at points inaccessible to other types of pyrometer. Both types are dependent on personal judgment and a certain degree of skill is required in their use. The choice between the two is largely a matter of individual preference, but where the radiation from a body is already polarised it may be more accurate to use the disappearing filament type.

For satisfactory use these instruments should be kept clean and handled with care. Both types are supplied already calibrated, but need periodic checking. The Wanner type can be recalibrated by means of the amyl acetate standard lamp and ground glass screen supplied with the instrument. It is important to use the screen having the same number as the instrument. It may also be checked against a black body furnace and thermocouple.

A more precise and quicker method of calibration is by means of a standard tungsten strip lamp. This method is briefly described below. The disappearing filament type pyrometer can only be checked by the standard strip lamp or by using a black body furnace with a thermocouple.

#### *Note on standard strip lamp for calibration*

Calibrated tungsten strip lamps can be purchased whose filament temperature is known for given power inputs. Provided a steady source of power is available, and can be measured accurately, a direct check can be made of an optical pyrometer over a wide temperature range.

#### D. PHOTO-ELECTRIC CELLS

These are being employed for certain purposes, *e.g.*, open-hearth roof pyrometry and will no doubt find increasing use in the future. Their application is similar to that of the total radiation pyrometer, but they are more sensitive in certain high temperature ranges.

#### E. PYROMETRIC CONES AND THERMOSCOPE BARS

*See Chapter I and Appendix 6.*

#### F. TEMPERATURE INDICATING PAINTS AND CRAYONS

At least two types of temperature indicating paints and crayons are available. The temperature is indicated by a colour change, which may be reversible or irreversible. Such indicators are useful for outside wall temperatures. The time factor is important in those indicators which have a colour change, and the data supplied by the manufacturers should be followed closely. Their accuracy can be checked by putting them on a metal strip and inserting the latter in a slotted metal block at known temperature.

## APPENDIX 4b

## TEMPERATURE—E.M.F. FOR THERMOCOUPLES

The following tables give typical data for the E.M.F.s. of chromel-alumel and platinum, platinum-rhodium thermocouples. The cold junction is assumed to be at 0°C. and a correction must therefore be made if other cold junction temperatures are employed. Since American Pt-Pt/Rh thermocouples appear to be different from those employed in Great Britain, two sets of tables are given. The British figures have been revised to take account of the 1948 International Temperature Scale. It should, however, be emphasised that there is always the risk of a slight batch-to-batch-variation in wire quality, and hence each new batch of couples should be checked against a standard couple or an absolute standard, *e.g.*, the melting points given in Appendix 5.

TEMPERATURE—E.M.F. VALUES FOR CHROMEL P-ALUMEL  
AND T<sub>1</sub>-T<sub>2</sub> ALLOY TYPES

Degree C.	0°	10°	20°	30°	40°	50°	60°	70°	80°	90°
0°	0.00	0.40	0.80	1.20	1.61	2.02	2.43	2.85	3.26	3.68
100°	4.10	4.51	4.92	5.33	5.73	6.13	6.53	6.93	7.33	7.73
200°	8.13	8.53	8.93	9.34	9.74	10.15	10.56	10.97	11.38	11.80
300°	12.21	12.62	13.04	13.45	13.87	14.29	14.71	15.13	15.55	15.97
400°	16.39	16.82	17.24	17.66	18.08	18.50	18.93	19.36	19.78	20.21
500°	20.64	21.07	21.49	21.92	22.34	22.77	23.20	23.62	24.05	24.48
600°	24.90	25.33	25.75	26.18	26.60	27.03	27.45	27.87	28.29	28.72
700°	29.14	29.56	29.98	30.40	30.82	31.23	31.65	32.07	32.48	32.90
800°	33.31	33.71	34.12	34.53	34.94	35.35	35.75	36.16	36.56	36.96
900°	37.36	37.76	38.16	38.56	38.96	39.35	39.75	40.14	40.55	40.92
1000°	41.31	41.70	42.09	42.48	42.87	43.25	43.63	44.02	44.40	44.78
1100°	45.16	45.54	45.92	46.29	46.67	47.07	47.41	47.78	48.15	48.52
1200°	48.89	49.25	49.62	49.98	50.34	50.69	51.05	51.41	51.76	52.11
1300°	52.46	52.81	53.16	53.51	53.85	54.20	54.54	54.88		

E.M.F. values are in millivolts (Absolute units 1948); temperatures are in ° Celsius. E.M.F. table is based on British Standards BS.1827; 1952.



## STEELPLANT REFRACTORIES

TEMPERATURE-E.M.F. VALUES FOR PLATINUM,  
 PLATINUM (87%)—RHODIUM (13%)  
 THERMOCOUPLES—AMERICAN

Degree C.	0°	10°	20°	30°	40°	50°	60°	70°	80°	90°
0°	0.00	0.06	0.12	0.18	0.25	0.31	0.38	0.45	0.52	0.60
100°	0.67	0.75	0.83	0.90	0.99	1.07	1.15	1.23	1.32	1.40
200°	1.49	1.58	1.67	1.76	1.85	1.94	2.03	2.12	2.21	2.30
300°	2.40	2.49	2.59	2.68	2.77	2.87	2.96	3.06	3.19	3.29
400°	3.40	3.51	3.61	3.72	3.82	3.93	4.04	4.15	4.25	4.36
500°	4.47	4.58	4.69	4.81	4.92	5.03	5.14	5.26	5.37	5.49
600°	5.60	5.72	5.83	5.95	6.06	6.18	6.30	6.42	6.53	6.65
700°	6.77	6.89	7.01	7.13	7.25	7.37	7.49	7.62	7.74	7.87
800°	7.99	8.12	8.24	8.37	8.49	8.62	8.75	8.88	9.00	9.13
900°	9.26	9.39	9.52	9.66	9.79	9.92	10.05	10.18	10.32	10.45
1000°	10.58	10.72	10.85	10.99	11.12	11.26	11.40	11.54	11.67	11.81
1100°	11.95	12.09	12.23	12.38	12.52	12.66	12.80	12.94	13.09	13.23
1200°	13.37	13.52	13.66	13.81	13.95	14.10	14.25	14.40	14.54	14.69
1300°	14.84	14.99	15.14	15.30	15.45	15.60	15.75	15.90	16.06	16.21
1400°	16.36	16.52	16.67	16.83	16.98	17.14	17.30	17.46	17.61	17.77
1500°	17.93	18.09	18.25	18.42	18.58	18.74	18.90	19.06	19.23	19.39
1600°	19.55	19.71	19.88	20.04	20.21	20.37	—	—	—	—

E.M.F. values are in millivolts; temperatures are in degrees centigrade.  
 (From values given in *Bulletin No. 325*, Charles Engelhard, Inc., New York.)

TEMPERATURE-E.M.F. VALUES FOR PLATINUM,  
 PLATINUM (87%)—RHODIUM (13%)  
 THERMOCOUPLES — BRITISH

Degree C.	0°	10°	20°	30°	40°	50°	60°	70°	80°	90°
0°	0.00	0.05	0.11	0.17	0.23	0.30	0.36	0.43	0.50	0.57
100°	0.64	0.72	0.80	0.88	0.96	1.04	1.12	1.20	1.29	1.38
200°	1.46	1.55	1.64	1.73	1.82	1.92	2.01	2.10	2.20	2.30
300°	2.39	2.49	2.59	2.69	2.79	2.89	2.99	3.09	3.19	3.29
400°	3.40	3.50	3.61	3.71	3.82	3.92	4.03	4.14	4.24	4.35
500°	4.46	4.57	4.68	4.79	4.90	5.01	5.12	5.23	5.35	5.46
600°	5.57	5.69	5.80	5.91	6.03	6.15	6.26	6.38	6.50	6.62
700°	6.74	6.86	6.98	7.10	7.22	7.34	7.46	7.58	7.71	7.83
800°	7.95	8.08	8.20	8.33	8.45	8.58	8.70	8.83	8.96	9.08
900°	9.21	9.34	9.47	9.59	9.72	9.85	9.98	10.11	10.25	10.38
1000°	10.51	10.64	10.78	10.91	11.04	11.18	11.31	11.45	11.58	11.72
1100°	11.85	11.99	12.12	12.26	12.40	12.53	12.67	12.81	12.95	13.08
1200°	13.22	13.36	13.50	13.64	13.78	13.92	14.06	14.20	14.34	14.48
1300°	14.62	14.76	14.90	15.04	15.18	15.33	15.47	15.61	15.75	15.90
1400°	16.04	16.18	16.33	16.47	16.61	16.75	16.90	17.04	17.18	17.32
1500°	17.46	17.60	17.75	17.89	18.03	18.17	18.30	18.44	18.58	18.72
1600°	18.86	18.99	19.13	19.26	19.40	19.53	19.67	19.80	19.94	20.07
1700°	20.20	20.33	20.47	20.60	20.73	20.86	20.99	—	—	—

E.M.F. values are in millivolts (Absolute units 1948). Temperatures are in degrees Celsius. E.M.F. table is based on British Standards BS.1826, 1952.

## APPENDIX 5

STANDARD FIXED TEMPERATURES USED FOR  
CALIBRATION OF THERMOCOUPLES  
AND PYROMETERS

<i>Substance</i>	<i>Point</i>	<i>Temperature* °C.</i>
Ice .. .. .	.. Melting ..	0
Water .. .. .	.. Boiling ..	100 (normal pressure)
Tin .. .. .	.. Freezing ..	231·9
Lead .. .. .	.. Freezing ..	327·4
Zinc .. .. .	.. Freezing ..	419·5
Sulphur .. .. .	.. Condensing ..	444·6
Aluminium .. .. .	.. Freezing ..	658·9
Sodium chloride .. .. .	.. Melting ..	800·4
Silver .. .. .	.. Freezing ..	960·5
Gold .. .. .	.. Freezing ..	1063
Diopside ( $\text{CaO} \cdot \text{MgO}_2 \cdot \text{SiO}_2$ ) .. .. .	.. Melting ..	1395
Nickel .. .. .	.. Melting ..	1450
Palladium .. .. .	.. Freezing ..	1552
Anorthite ( $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) .. .. .	.. Melting ..	1552
Platinum .. .. .	.. Melting ..	1750
Tungsten .. .. .	.. Melting ..	3345

\* 1948 International Temperature Scale.

## APPENDIX 6a

TEMPERATURE END POINTS OF  
PYROMETRIC CONES

Apart from being used in the laboratory, pyrometric cones are employed quite extensively for control of kiln temperatures in the firing of refractory materials.

Thermoscope bars are also used to a limited extent for kiln control and a table showing the bending temperatures of the type of bars used in Great Britain is included in Appendix 6b.

## APPENDIX 6a—continued

Cone number				End point			
				American standard*		Seger cones†	
				°C.	°F.	°C.	°F.
1	..	..	..	1160	2120	(a) 1100	2012
2	..	..	..	1165	2129	(a) 1120	2048
3	..	..	..	1170	2138	(a) 1140	2084
4	..	..	..	1190	2174	(a) 1160	2120
5	..	..	..	1205	2201	(a) 1180	2156
6	..	..	..	1230	2246	(a) 1200	2192
7	..	..	..	1250	2282	1230	2246
8	..	..	..	1260	2300	1250	2282
9	..	..	..	1285	2345	1280	2336
10	..	..	..	1305	2381	1300	2372
11	..	..	..	1325	2417	1320	2408
12	..	..	..	1335	2435	1350	2462
13	..	..	..	1350	2462	1380	2552
14	..	..	..	1400	2552	1410	2570
15	..	..	..	1435	2615	1435	2615
16	..	..	..	1465	2669	1460	2660
17	..	..	..	1475	2687	1480	2696
18	..	..	..	1490	2714	1500	2732
19	..	..	..	1520	2768	1520	2768
20	..	..	..	1530	2786	1530	2786
23	..	..	..	1580	2876	—	—
26	..	..	..	1595	2903	1580	2876
27	..	..	..	1605	2921	1610	2930
28	..	..	..	1615	2939	1630	2966
29	..	..	..	1640	2984	1650	3002
30	..	..	..	1650	3002	1670	3038
31	..	..	..	1680	3056	1690	3074
32	..	..	..	1700	3092	1710	3110
32½	..	..	..	1722	3131	—	—
33	..	..	..	1745	3173	1730	3146
34	..	..	..	1760	3200	1750	3182
35	..	..	..	1785	3245	1770	3218
36	..	..	..	1810	3290	1790	3254
37	..	..	..	1820	3308	1825	3317
38	..	..	..	1835	3335	1850	3362
Normal heating rate ..				1 to 20=150°C. per hr. 23 to 38=100°C. " "		600°C. per hr.	

\* FAIRCHILD and PETERS, *J. Amer. Ceram. Soc.*, 1926; 9, 701-743, *A.S.T.M. Manual on Refractory Materials*, 1952.

† INSTITUTION OF GAS ENGINEERS, *Standard Specification for Refractory Materials*, 1934.

## APPENDIX 6b

APPROXIMATE BENDING TEMPERATURES OF  
HOLDCROFT'S THERMOSCOPE BARS

<i>Bar No.</i>	<i>°C.</i>	<i>°F.</i>	<i>Bar No.</i>	<i>°C.</i>	<i>°F.</i>
1	600	1112	22	1080	1976
2	650	1202	23	1100	2012
3	670	1238	24	1120	2048
4	700	1292	25	1140	2084
5	730	1346	26	1200	2192
6	760	1400	26a	1230	2246
7	790	1454	27	1250	2282
7a	810	1490	27a	1270	2318
8	840	1544	28	1280	2336
9	860	1580	29	1300	2372
10	875	1607	30	1325	2417
11	890	1634	31	1350	2462
12	905	1661	32	1380	2516
13	920	1688	33	1430	2606
14	935	1715	34	1460	2660
15	950	1742	35	1475	2687
16	960	1760	36	1490	2714
17	970	1778	37	1505	2741
18	985	1805	38	1520	2768
19	1000	1832	39	1535	2795
20	1040	1904	40	1550	2822
21	1060	1940			

## APPENDIX 7

## COLOUR TEMPERATURE CHARTS

Various colour-temperature charts have been suggested and 4 such charts are compared in the diagram below. The differences emphasise the danger of using colour as more than a rough guide to furnace temperature.

Temperature °C.	A	B	C	D
400				
500	Red—Just visible.	Earliest Visible	Incipient Red Heat.	Lowest Visible Red to
600		Red		Dark Red.
700	Dull Red.		Dark Red Heat.	Dark to Cherry Red.
800	Dull Cherry Red.	Dull Red.		Cherry to Bright Cherry Red.
900	Cherry Red. Bright Cherry Red.	Full Red.	Bright Red Heat.	Bright Cherry to Orange.
1000	Orange (deep).	Bright Red.		Orange to Yellow
1100	Orange (clear).	Orange.	Yellowish Red Heat.	
1200	Light Yellow.	Bright Orange or Yellow.		Yellow to Light Yellow.
1300	White.		Incipient White Heat.	
1400	Bright White.			Light Yellow to
1500	Dazzling White.	White.	White Heat.	White.
1600		Dazzling White.		Dazzling White.

For a more precise approach see:

D. B. JUDD, *J. Res. Nat. Bur. Stand.*, 1950, 44, 1.

H. G. W. HARDING, *Proc. Physical Soc.*, 1950, B, 63, 685.

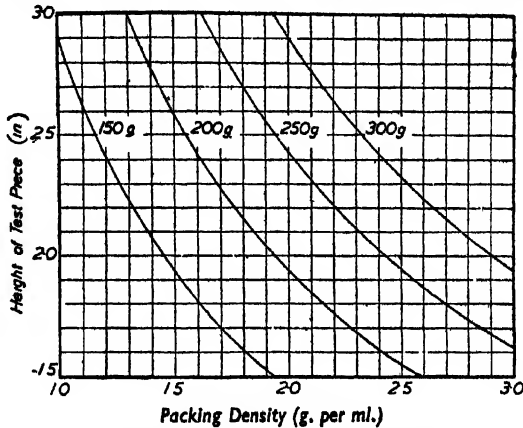
# APPENDIX 8 COMPARISON OF STANDARD SIEVE SERIES

U.S. STANDARDS				TYLER STANDARDS				BRITISH STANDARDS			
Sieve No.	Sieve opening in.	Sieve opening mm.	Wire diameter in.	Mash	Sieve opening in.	Sieve opening mm.	Wire diameter in.	Mash No.	Sieve opening in.	Sieve opening mm.	Wire diameter in.
2½	0.315	8.0	0.073	2½	1.050	26.67	0.148	5	½ in.	12.70	0.128
3	0.265	6.73	0.065	3	0.742	18.85	0.135	6	⅝ in.	9.53	0.104
3½	0.223	5.66	0.057	3½	0.545	13.93	0.105	7	¾ in.	6.35	0.092
4	0.187	4.76	0.050	4	0.371	9.423	0.088	8	⅞ in.	4.76	0.080
5	0.157	4.00	0.044	5	0.263	7.925	0.070				
6	0.132	3.36	0.040	6	0.221	5.613	0.065				
7	0.111	2.83	0.036	7	0.185	4.699	0.065				
8	0.0937	2.38	0.031	8	0.156	3.962	0.044				
10	0.0787	2.00	0.0331	10	0.131	3.327	0.036				
12	0.0661	1.68	0.0299	12	0.110	2.794	0.033				
14	0.0555	1.41	0.0272	14	0.093	2.362	0.032				
16	0.0469	1.19	0.0240	16	0.078	1.981	0.033				
18	0.0394	1.00	0.0213	18	0.065	1.651	0.035				
20	0.0331	0.84	0.0189	20	0.055	1.397	0.028				
25	0.0280	0.71	0.0165	25	0.046	1.168	0.025				
30	0.0232	0.59	0.0146	30	0.039	0.991	0.0235				
35	0.0197	0.50	0.0130	35	0.0328	0.833	0.0172				
40	0.0165	0.42	0.0114	40	0.0276	0.701	0.0141				
45	0.0138	0.35	0.0098	45	0.0232	0.589	0.0125				
50	0.0117	0.297	0.0087	50	0.0195	0.495	0.0118				
60	0.0098	0.250	0.0074	60	0.0164	0.417	0.0122				
70	0.0083	0.210	0.0064	70	0.0138	0.351	0.0100				
80	0.0070	0.177	0.0055	80	0.0116	0.295	0.0092				
100	0.0059	0.149	0.0047	100	0.0097	0.246	0.0070				
120	0.0049	0.125	0.0040	120	0.0082	0.208	0.0056				
140	0.0041	0.105	0.0034	140	0.0069	0.175	0.0042				
170	0.0035	0.088	0.0029	170	0.0058	0.147	0.0038				
200	0.0029	0.074	0.0025	200	0.0049	0.124	0.0034				
230	0.0024	0.062	0.0021	230	0.0041	0.104	0.0026				
270	0.0021	0.053	0.0018	270	0.0035	0.088	0.0024				
325	0.0017	0.044	0.0016	325	0.0029	0.074	0.0021				
					0.0024	0.061	0.0016				
					0.0021	0.053	0.0016				
					0.0017	0.043	0.0014				

## APPENDIX 9

## A.F.A. RAMMER CALIBRATION CURVES

The packing density of standard A.F.A. test pieces of 150, 200, 250, or 300 g. weight can be conveniently determined from the diagram given below. When determining the packing density of dry aggregates it is useful to calibrate the A.F.A. rammer and measure the length of the test piece indirectly, using the top end of the plunger shaft and a depth gauge.



*Relation between height and packing density of A.F.A. test pieces.*



## PROPERTIES OF MINERALS ASSOCIATED

Name	Synonym	Formula	Specific gravity	Hardness
Akermanite ..	—	$2\text{CaO} \cdot \text{MgO} \cdot 2\text{SiO}_2$ ..	3.12-3.18	—
Albite ..	Sodium felspar ..	$\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ..	2.61-2.64	6.0-6.5
Anatase ..	Octahedrite ..	$\text{TiO}_2$ ..	3.82-3.95	5.5-6.0
Andalusite ..	—	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ..	3.1-3.2	7.0-7.5
Anorthite ..	Lime felspar ..	$\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ..	2.703-2.763	6.0-6.5
Anorthoclase ..	Soda microcline ..	$(\text{Na}, \text{K})_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ..	2.56-2.65	6.0-6.5
Antigorite ..	—	$3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ..	2.55-2.62	3-4
Aragonite ..	—	$\text{CaCO}_3$ ..	2.85-2.94	3.5-4.0
Augite ..	—	$\text{CaMg}(\text{SiO}_3)_2 + (\text{Mg}, \text{Fe}) - (\text{Al}, \text{Fe})_2\text{SiO}_6$ ..	3.2-3.6	5-6
Baddeleyite ..	—	$\text{ZrO}_2$ ..	5.50-6.03	6.5
Bauxite ..	—	$\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ ..	2.55	1-3
Beryllia ..	Brommelite ..	$\text{BeO}$ ..	3.0	—
Biotite ..	Black mica ..	$(\text{K}, \text{H})_2(\text{Mg}, \text{Fe})_3(\text{Al}, \text{Fe})_3(\text{SiO}_3)_9$ ..	2.69-3.16	2.5-3.0
Borax ..	Tincal ..	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ..	1.69-1.72	2.0-2.5
Brookite ..	—	$\text{TiO}_2$ ..	3.87-4.08	5.5-6.0
Brownmillerite ..	—	$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ ..	3.77	—
Brucite ..	—	$\text{MgO} \cdot \text{H}_2\text{O}$ ..	2.38-2.4	2.5
Calcite ..	Calcspar, Iceland spar	$\text{CaCO}_3$ ..	2.711	3
Calcium ferrite	—	$\text{CaO} \cdot \text{Fe}_2\text{O}_3$ ..	—	—
Calcium fluoride	Fluorspar, fluorite ..	$\text{CaF}_2$ ..	3.18	—
Chalcedony ..	Flint, agate ..	$\text{SiO}_2$ ..	2.55-2.63	6
Chromic oxide ..	—	$\text{Cr}_2\text{O}_3$ ..	5.2	—
Chromite ..	—	$\text{FeO} \cdot \text{Cr}_2\text{O}_3$ ..	4.32-4.57	5.5
Clinoenstatite ..	—	$\text{MgO} \cdot \text{SiO}_2$ ..	3.19-3.28	—
Cordierite a ..	—	$2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$ ..	2.77	—
Corundum ..	Ruby, sapphire ..	$\text{Al}_2\text{O}_3$ ..	3.95-4.10	9
Cristobalite ..	—	$\text{SiO}_2$ ..	2.32	6-7
Cyanite ..	Kyanite ..	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ..	3.559-3.675	4-7
Diaspore ..	—	$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ..	3.3-3.5	6.5-7.0
Dicalcium ferrite	—	$2\text{CaO} \cdot \text{Fe}_2\text{O}_3$ ..	—	—
Dicalcium silicate	—	$2\text{CaO} \cdot \text{SiO}_2$ (three forms) $\begin{cases} \alpha & 3.27 \\ \beta & 3.28 \\ \gamma & 2.97 \end{cases}$	3.27 3.28 2.97	5.6 — —
Diopside ..	Malacolite, alalite ..	$\text{CaMg}(\text{SiO}_3)_2$ ..	3.20-3.38	5-6
Dolomite ..	Pearl spar ..	$\text{CaCO}_3 \cdot \text{MgCO}_3$ ..	2.80-2.99	3.5-4.5
Enstatite ..	+ Fe, bronzite, hypersthene	$\text{MgO} \cdot \text{SiO}_2$ ..	3.10-3.43	5-6
Fayalite ..	—	$\text{Fe}_2\text{SiO}_4$ ..	3.91-4.34	6.5
Forsterite ..	—	$\text{Mg}_2\text{SiO}_4$ ..	3.191-3.33	6-7
Gehlenite ..	—	$2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ..	2.9-3.07	5.5-6
Gibbsite ..	Hydrargillite ..	$\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ..	2.3-2.42	2.5-3.5
Graphite ..	Plumbago, black lead	C (traces of Fe, $\text{SiO}_2$ , etc.) ..	2.09-2.25	1-2
Gypsum ..	—	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ..	2.314-2.328	1.5-2
Hematite ..	—	$\text{Fe}_2\text{O}_3$ ..	4.9-5.3	5.5-6.5
Hercynite ..	Iron spinel ..	$\text{FeO} \cdot \text{Al}_2\text{O}_3$ ..	3.91-3.95	7.5-8
Ilmenite ..	Menaccinite, titanite iron ore	$\text{FeO} \cdot \text{TiO}_2$ ..	4.44-4.90	5-6

## DIX IO

## WITH REFRACTORIES AND SLAGS

<i>Name</i>	<i>Crystalline form and colour (natural or artificial)</i>	<i>Index of refraction</i>	<i>Melting or dissociation point (°C.)</i>
Akermanite ..	Tetr., colourless .. ..	1·632; 1·639 .. ..	1458
Albite ..	Triclinic., grey, or rarely coloured ..	1·525; 1·529; 1·536 .. ..	1100
Anatase ..	Tetra., br., bl., blk. .. ..	2·554; 2·493 .. ..	—
Andalusite ..	Rhomb., grey., redsh., grnsh., blsh. ..	1·632; 1·638; 1·643 .. ..	1810
Anorthite ..	Tricl., col., wh., or greyish. .. ..	1·575; 1·583; 1·588 .. ..	1550
Anorthoclase ..	Tricl. .. ..	1·523; 1·529; 1·531 .. ..	—
Antigorite ..	Rhomb., brnsh., green .. ..	1·56; 1·57 .. ..	1557
Aragonite ..	Rhomb., col., wh., yel., redsh., blsh., blk. .. ..	1·530; 1·681; 1·685 .. ..	—
Augite ..	Monocl., dk. grn. to blk. .. ..	1·712; 1·717; 1·733 .. ..	—
Baddeleyite ..	Monocl., col.-yel., br., blk. .. ..	2·13; 2·19; 2·20 .. ..	ca.2700
Bauxite ..	Amor., wh., br., yel., or redsh. .. ..	1·570 .. ..	—
Beryllia ..	Hexagonal, colourless .. ..	1·719; 1·733 .. ..	ca.2500
Biotite ..	Monocl., black or dk. br. or grn. .. ..	1·541; 1·574; 1·574 .. ..	—
Borax ..	Monocl., wh., greyish, blsh., or grnsh. ..	1·447; 1·469; 1·471 .. ..	—
Brookite ..	Rhomb., br., yelsh., redsh. to iron blk. ..	2·583; 2·586; 2·741 .. ..	1900
Brownmillerite ..	Rhomb., redsh. br. .. ..	1·96; 2·01; 2·04 .. ..	1415
Brucite ..	Trig., wh., grey, blue, or grn. .. ..	1·56; 1·58 .. ..	—
Calcite ..	Hex. col., wh., or yelsh. .. ..	1·658; 1·486 .. ..	—
Calcium ferrite ..	Rhomb. or uniaxial., black-red .. ..	2·43; 2·58 .. ..	1216
Calcium fluoride ..	Cubic, colourless .. ..	1·434 .. ..	1340
Chalcodony ..	Wh., greyish bl., br.-blk. .. ..	1·537; (1·533-1·539); 1·530 ..	1710
Chromic oxide ..	Hexagonal, green .. ..	ca.2·5 .. ..	2150-2430
Chromite ..	Cub., blk.-brnsh. blk. .. ..	2·07-2·16 .. ..	2180
Clinocostatite ..	Monocl., colourless .. ..	1·65; 1·64; 1·66 .. ..	1557
Cordierite a ..	Rhomb., col. .. ..	1·52; 1·52-1·58 .. ..	1550
Corundum ..	Hex. (trig. rhodr.), col., red., yel., bl., br., or grey .. ..	1·768; 1·760 .. ..	2050
Cristobalite ..	Tetragonal .. ..	1·484; 1·487 .. ..	1710
Cyanite ..	Tricl., bl., grey, wh., grn., or blk. .. ..	1·712; 1·720; 1·728 .. ..	1810
Diaspore ..	Rhomb., grey., wh., pink., yel., br. .. ..	1·702; 1·722; 1·750 .. ..	—
Dicalcium ferrite ..	Blk., yel.-br. .. ..	2·25-2·27 .. ..	1436
Dicalcium silicate ..	Monocl., or tricl., col. .. ..	1·715; 1·720; 1·737 .. ..	2130
Diopside ..	Monocl. (?) .. ..	1·717; 1·735 .. ..	—
Dolomite ..	Monocl., lt. to dk. grn., col., grey., yel., rar., bl. .. ..	1·642; 1·645; 1·654 .. ..	—
Enstatite ..	Monocl., it. to dk. grn., col., grey., yel., rar., bl. .. ..	1·664; 1·671; 1·694 .. ..	1391
Fayalite ..	Hex. (trig. rhodr.), wh., yel., redsh., br., blk. .. ..	1·682; 1·503 .. ..	—
Forsterite ..	Rhomb., greyish or yelsh., wh., grnsh., or brnsh. .. ..	1·650; 1·653; 1·658 .. ..	1557
Gehlenite ..	Rhomb., yel.-blk. .. ..	1·835; 1·877; 1·886 .. ..	1205
Gibbsite ..	Rhomb., wh., grnsh., yelsh. .. ..	1·635; 1·651; 1·670 .. ..	1890
Graphite ..	Tetr., greyish, grn., to brn. .. ..	1·669; 1·658 .. ..	1590
Gypsum ..	Monocl., wh., grnsh., redsh., to yel. ..	1·566; 1·566; 1·587 .. ..	—
Hematite ..	Hex., blk., dk. grey .. ..	Opaque .. ..	—
Hercynite ..	Monocl., wh., oft. yel., red., br., blk. ..	1·520; 1·523; 1·530 .. ..	—
Ilmenite ..	Hex. (trig), st. grey-blk. .. ..	(Li): 3·01; 2·94 .. ..	—
	Cub. blk. .. ..	1·800 .. ..	> 1750
	Hex. (trig) iron-brnsh. black .. ..	Opaque .. ..	—

## PROPERTIES OF MINERALS ASSOCIATED

<i>Name</i>	<i>Synonym</i>	<i>Formula</i>	<i>Specific gravity</i>	<i>Hardness</i>
Kaolinite ..	China clay, kaolin ..	$\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ..	2.60-2.63	2.0-2.5
Labradorite ..	Labrador felspar int. bet. albite and anorthite	$\text{NaAlSi}_3\text{O}_8$ , $\text{CaAl}_2\text{Si}_2\text{O}_8$ ratio 1 : 1 to 1 : 3	2.70-2.72	5.0-6.0
Lime ..	—	$\text{CaO}$ .. .. .	3.32	—
Magnesite ..	— ..	$\text{MgCO}_3$ .. .. .	2.95-3.2	3.5-4.5
Magnetite ..	Lodestone ..	$\text{Fe}_3\text{O}_4$ .. .. .	4.967-5.180	5.5-6.5
Melilite ..	—	$\text{Na}_2(\text{Ca}, \text{Mg})_{11}(\text{Al}, \text{Fe})_4(\text{SiO}_4)_8$	2.9-3.4	5-6
Merwinite ..	—	$3\text{CaO} \cdot \text{MgO} \cdot 2\text{SiO}_2$ ..	3.15	—
Microcline ..	—	$\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ..	2.54-2.57	6.0-6.5
Monticellite ..	—	$\text{CaMgSiO}_4$ .. .. .	3.03-3.25	5.0-5.5
Muscovite ..	White mica ..	$\text{K}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ..	2.76-3.00	2.5-3.0
Mullite ..	—	$3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ .. ..	3.03	—
Nephelite ..	Nepheline, elaeolite ..	$(\text{Na}, \text{K})_3\text{Al}_3\text{Si}_3\text{O}_{24}$ or $\text{NaAlSiO}_4$ .. .. .	2.55-2.65	5.5-6.0
Oligoclase ..	Lime-soda Felspar ..	$\text{NaAlSi}_3\text{O}_8 + \text{CaAl}_2\text{Si}_2\text{O}_8$	2.62-2.672	6-7
Olivine ..	Chrysolite, peridot ..	$(\text{Mg}, \text{Fe})_2\text{SiO}_4$ .. ..	3.26-3.40	6.5-7.0
Opal ..	—	$\text{SiO}_2 \cdot x\text{H}_2\text{O}$ .. ..	2.1-2.3	5.5-6.5
Orthoclase ..	Potassium felspar ..	$\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ .. ..	2.56	6
Periclase ..	—	$\text{MgO}$ .. .. .	3.58-3.67	5.5-6
Picotite ..	Chrome-spinel ..	$(\text{Mg}, \text{Fe})\text{O} \cdot (\text{Al}, \text{Cr})_2\text{O}_3$	4.08	—
Pyrite ..	Iron pyrites, fool's gold	$\text{FeS}_2$ .. .. .	4.95-5.17	6.0-6.5
Quartz ..	—	$\text{SiO}_2$ .. .. .	2.65	7
Rhodonite ..	Fowlerite ..	$\text{MnO} \cdot \text{SiO}_2$ .. ..	3.40-3.68	5.5-6.5
Rutile ..	Nigrine ..	$\text{TiO}_2$ .. .. .	4.18-4.25	6.0-6.5
Serpentine ..	Incl. chrysotile or asbestos ..	$3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ..	2.50-2.65	2.5-4.0
Siderite ..	Spathic iron, chalybite	$\text{FeCO}_3$ .. .. .	3.83-3.88	3.5-4.5
Silicon carbide ..	—	$\text{SiC}$ .. .. .	3.2	—
Sillimanite ..	Fibrolite ..	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ .. ..	3.23-3.25	6.0-7.5
Spessartite ..	Manganese aluminium garnet ..	$3\text{MnO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ ..	4.0-4.3	—
Spinel ..	—	$\text{MgO} \cdot \text{Al}_2\text{O}_3$ .. ..	3.5-4.1	8
Talc ..	Soapstone, steatite ..	$3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ ..	2.7-2.8	1.0-1.5
Tephroite ..	—	$\text{Mn}_2\text{SiO}_4$ .. .. .	4.04	6
Thoria ..	Thorianite ..	$\text{ThO}_2$ .. .. .	9.7-9.8	—
Tri-calcium silicate ..	—	$3\text{CaO} \cdot \text{SiO}_2$ .. ..	3.15	—
Tridymite ..	—	$\text{SiO}_2$ .. .. .	2.26-2.28	7
Wollastonite ..	—	$\text{CaO} \cdot \text{SiO}_2$ .. ..	2.80-2.92	4.5-5.0
Zircon ..	Hyacinth, jargoon ..	$\text{ZrSiO}_4$ .. .. .	4.02-4.86	7.5

## DIX 10—continued

## WITH REFRACTORIES AND SLAGE—continued

<i>Name</i>	<i>Crystalline form and colour (natural or artificial)</i>	<i>Index of refraction</i>	<i>Melting or dissociation point (°C.)</i>
Kaolinite ..	Monocl., wh., yelsh., redsh., blsh., grnsh., brnsh. .. ..	1·561; 1·565; 1·566 .. ..	—
Labradorite ..	Tricl., grey, br., or grnsh .. ..	1·559; 1·563; 1·568 .. ..	1280-1300
Lime ..	Cubic, colourless .. ..	1·838 .. ..	2570
Magnesite ..	Hex. (trig.) col., wh.-yelsh., .. br.-blk. .. ..	1·700; 1·509 .. ..	—
Magnetite ..	Cub. iron blk. .. ..	2·42 .. ..	1590
Melilite ..	Tetr., wh., yel., grnsh., redsh., br. ..	1·634; 1·639 .. ..	—
Merwinite ..	Monocl. .. ..	1·708; 1·711; 1·724 .. ..	1590
Microcline ..	Tricl., wh., yelsh., grey, grn., or red	1·522; 1·526; 1·530 .. ..	1530
Monticellite ..	Rhomb., col. to grey .. ..	1·64; 1·65; 1·65 .. ..	—
Muscovite ..	Monocl. col. or pa. yel., grey or br., rare rose .. ..	1·561; 1·590; 1·594 .. ..	—
Mullite ..	Rhomb., col., pink, bl. .. ..	1·642; 1·644; 1·654 .. ..	1810
Nephelite ..	Hex., col., wh., yelsh., grey or red ..	1·537; 1·533 .. ..	1526
Oligoclase ..	Tricl., wh., grey, grnsh., redsh. ..	1·539; 1·543; 1·547 .. ..	1120-1380
Olivine ..	Rhomb., olv. grn., or greyish grn. to yelsh. br. .. ..	1·662; 1·680; 1·699 .. ..	—
Opal ..	Amor., col., wh., yel., br., red, grn., int. refl. .. ..	1·41-1·46 .. ..	—
Orthoclase ..	Moncl., col., wh., pa. yel., flesh red to grey .. ..	1·518; 1·524; 1·526 .. ..	—
Periclase ..	Cub., col. .. ..	1·736 .. ..	2800
Picotite ..	Cub. yelsh. br., grnsh. br. to blk. ..	2·05 .. ..	—
Pyrite ..	Cub. pa. brass to gold yel. .. ..	Opaque .. ..	—
Quartz ..	Hex. (trig.), col. or yel., rose, br., grn., bl., grey .. ..	1·544; 1·553 .. ..	1710
Rhodonite ..	Tricl., red, pink, yelsh., grnsh., brnsh., blk. .. ..	1·733; 1·737; 1·744 .. ..	1273
Rutile ..	Tetra., redsh. br., red, yelsh., blsh., vit., blk. .. ..	2·616; 2·903 .. ..	1825
Serpentine ..	Monocl. (opt.), fibrous var., asbestos; grey to grnsh. or brnsh. .. ..	1·490-1·571 .. ..	1557
Siderite ..	Hex., brnsh. to blk., grey, grn., wh. ..	1·872; 1·634 .. ..	—
Silicon carbide ..	Hex., col., green, blue, black .. ..	2·697; 2·654 .. ..	3400
Sillimanite ..	Rhomb., grey, br., yelsh., grnsh ..	1·66; 1·66; 1·68 .. ..	1810
Spessartite ..	Cub. dk. red to brnsh. red .. ..	1·811 .. ..	1200
Spinel ..	Cub. col. or red, bl., grn., yel., br., blk. .. ..	1·723 .. ..	2135
Talc ..	Moncl., wh., grnsh., wh., lt. grn. ..	1·539; 1·589; 1·589 .. ..	1543
Tephroite ..	Rhomb., grey to rose red .. ..	1·78; 1·80; 1·82 .. ..	1290-1320
Thoria ..	Cubic, colourless .. ..	ca.2·20 .. ..	3050
Tri-calcium silicate ..	Col. .. ..	1·718; 1·723 .. ..	1900
Tridymite ..	Rhomb., col. or wh. .. ..	1·469; 1·47; 1·473 .. ..	1710
Wollastonite ..	Monocl., wh.-grey, yel., red or br. ..	1·61-1·65 .. ..	1540
Zircon ..	Tetr., col., pa. yel., grey., yelsh. grn., br. redsh. br. .. ..	1·93; 1·99 .. ..	2550

## APPENDIX II

## ABSOLUTE DENSITY OF WATER

Density in grams per cubic centimetre, computed from the relative values by Thiesen, Scheel, and Disselhorst (1900), and the absolute value at 3.98°C. by the International Bureau of Weights and Measures (1910).

Degrees	0	1	2	3	4	5	6	7	8	9
0	0.999841	847	854	860	866	872	878	884	889	895
1	900	905	909	914	918	923	927	930	934	938
2	941	944	947	950	953	955	958	960	962	964
3	965	967	968	969	970	971	972	972	973	973
4	973	973	973	972	972	972	970	969	968	966
5	965	963	961	959	957	955	952	950	947	944
6	941	938	935	931	927	924	920	916	911	907
7	902	898	893	888	883	877	872	866	861	855
8	849	843	837	830	824	817	810	803	796	789
9	781	774	766	758	751	742	734	726	717	709
10	700	691	682	673	664	654	645	635	625	615
11	605	595	585	574	564	553	542	531	520	509
12	498	486	475	463	451	439	427	415	402	390
13	377	364	352	339	326	312	299	285	272	258
14	244	230	216	202	188	173	159	144	129	114
15	099	084	069	054	038	023	007	991	975	959
16	0.998943	926	910	893	877	860	843	826	809	792
17	774	757	739	722	704	686	668	650	632	613
18	595	576	558	539	520	501	482	463	444	424
19	405	385	365	345	325	305	285	265	244	224
20	203	183	162	141	120	099	078	056	035	013
21	0.997992	970	948	926	904	882	860	837	815	792
22	770	747	724	701	678	655	632	608	585	561
23	538	514	490	466	442	418	394	369	345	320
24	296	271	246	221	196	171	146	120	095	069
25	044	018	992	967	941	914	888	862	836	809
26	0.996783	756	729	703	676	649	621	594	567	540
27	512	485	457	429	401	373	345	317	289	261
28	232	204	175	147	118	089	060	031	002	973
29	0.995944	914	885	855	826	796	766	736	706	676
30	646	616	586	555	525	494	464	433	402	371

## APPENDIX 12

## MOHS' SCALE OF HARDNESS\*

- |                        |                          |
|------------------------|--------------------------|
| 1. Talc                | 6. Orthoclase (feldspar) |
| 2. Gypsum or rock salt | 7. Quartz                |
| 3. Calcite             | 8. Topaz                 |
| 4. Fluorite            | 9. Corundum              |
| 5. Apatite             | 10. Diamond              |

Hardness is one of the properties used in describing and identifying minerals. No absolute standards are available, but Mohs' scale affords a useful guide. The ten minerals are arranged in order of increasing hardness, and any other mineral can be given a hardness number according as to whether it does or does not scratch the above minerals. Thus periclase (MgO) has a Mohs' hardness of 5-6, since it scratches apatite, but is scratched by orthoclase.

\**Dana's Textbook on Mineralogy*, J. Wiley & Sons, Inc., New York, 1932.

## APPENDIX 13

## THERMAL EXPANSION DATA AND EXPANSION ALLOWANCES

The following expansion data are typical for various types of refractories:

	Per cent. expansion (20°-1000°C.)
Fireclay brick .. .. .	0.5-0.7
Forsterite brick .. .. .	1.1
Chrome brick .. .. .	0.8-0.9
Chrome-magnesite brick ..	0.8-0.9
Stabilised dolomite brick ..	1.3
Magnesite brick .. .. .	1.3-1.4
Silica brick .. .. .	1.2-1.4

In applying these data, it must be remembered that the thermal expansion of silica is not linear (*see* Chapter II).

The following expansion allowances have been recommended for building structures:

	$\frac{1}{16}$ - $\frac{3}{32}$ in. per foot
Fireclay brick .. .. .	$\frac{1}{16}$ " " "
Forsterite brick .. .. .	$\frac{1}{16}$ " " "
Chrome brick .. .. .	$\frac{3}{32}$ " " "
Chrome-magnesite brick ..	$\frac{3}{16}$ " " "
Magnesite brick .. .. .	$\frac{1}{4}$ " " "
Silica brick .. .. .	$\frac{1}{8}$ - $\frac{3}{16}$ " " "

The true expansion allowance for a refractory structure will, of course, depend on the working temperature in addition to the expansion characteristics of the material used. There are not sufficient data available to lay down hard and fast rules for this, but the figures given above provide a rough guide for expansion allowances.

## PROPERTIES OF SELECTED

Brick type	Chemical analysis  per cent.		Apparent Porosity  %	Bulk density		Refractoriness  °C.	Cold crushing strength (lb./sq. in.)	Refractoriness-under-load		Permanent linear change on reheating
				g./ml.	lb./cu. ft.			Rising temp. test 50 lb./sq. in.	Maintained temp. test 25 lb./sq. in.	
Siliceous Fireclay	SiO <sub>2</sub>	63.9	23.7	2.00	125	1550	6910	Initial deformation 1070°C. Fail 1520°C.	—	2 hours at 1410°C. 3.6 per cent. (Exp.)
	Al <sub>2</sub> O <sub>3</sub>	27.5								
	Fe <sub>2</sub> O <sub>3</sub>	5.1								
	TiO <sub>2</sub>	0.9								
	CaO	0.5								
	MgO	0.8								
	Alkalis	1.1								
Normal Fireclay	SiO <sub>2</sub>	53.6	21.7	1.98	123	1680	4140	Initial deformation 1220°C. Fail 1540°C.	—	2 hours at 1410°C. 0.6 per cent. (Con.)
	Al <sub>2</sub> O <sub>3</sub>	36.8								
	Fe <sub>2</sub> O <sub>3</sub>	5.7								
	TiO <sub>2</sub>	1.1								
	CaO	0.7								
	MgO	1.0								
	Alkalis	0.9								
Silica (hard-fired -S.G.2.31)	SiO <sub>2</sub>	95.6	25.4	1.73	108	1710	*4680	—	1600°C. for 1 hour 0.6 per cent. collapse	2 hours 1500°C. 0.2 per cent. (Exp.)
	Al <sub>2</sub> O <sub>3</sub>	0.6								
	Fe <sub>2</sub> O <sub>3</sub>	1.1								
	TiO <sub>2</sub>	0.1								
	CaO	2.1								
Semi-silica (sand)	SiO <sub>2</sub>	88.9	23.6	1.93	121	*1630	*1480	*25 lb./sq. in. load. Initial deformation 1450°C. Fail 1520°C.	—	Approximately volume stable
	Al <sub>2</sub> O <sub>3</sub>	9.1								
	Fe <sub>2</sub> O <sub>3</sub>	1.0								
	TiO <sub>2</sub>	0.4								
	CaO	0.2								
	MgO	0.3								
	Alkalis	0.4								

\* Properties were obtained on bricks of similar type, but not on bricks taken from the same batch.

# DIX 14

## REFRACTORY BRICKS

<i>Thermal shock resistance</i>	<i>Resistance to slags</i>	<i>Thermal expansion (20°-1000°C.)</i>	<i>Thermal conductivity</i>	<i>Specific heat</i>	<i>Comments</i>
Moderate	Moderate resistance to acid and basic open-hearth slags, coal ash, iron oxide, etc.	*0.5%	*About 9 B.Th.U. —500°C. mean	*0.25	A good general purpose brick for use in the construction of reheating furnaces, ladle linings, etc., where the relatively low refractoriness is no disadvantage.
Excellent	Good resistance to acid and basic open-hearth slags, coal ash, iron oxide, etc.	0.75%	*About 9 B.Th.U. —500°C. mean	*0.25	An excellent general purpose brick for use in reheating furnaces, open-hearth furnace doors and checkers, soaking pits, boilers, etc.
Low, especially up to 300°C.	High resistance to acid slags and remarkable tolerance for iron oxide	*1.25% (irregular)	*10 B.Th.U. —500°C. mean	*0.26	Hard-fired silica brick of the type recommended for the linings and roofs of open-hearth furnaces, upper courses of checkers, coke ovens, etc.
Moderate	Moderately good resistance to slags high in iron and lime, e.g., open-hearth furnace checker dust	*0.66% (irregular)	6 B.Th.U. —500°C. mean	0.26	Suitable for use in the lower courses of open-hearth furnace checkers and in reheating furnaces. Particularly useful where volume stability is essential. Glazes well in the presence of coal ash.



APPEN  
PROPERTIES OF SELECTED

Brick type	Chemical analysis  per cent.	Apparent Porosity  %	Bulk density		Refractoriness  °C.	Cold crushing strength (lb./sq. in.)	Refractoriness-under-load		Permanent linear change on reheating
			g./ml.	lb./cu. ft.			Rising temp. test 50 lb./sq. in.	Maintained temp. test 25 lb./sq. in.	
Magnesite (Spalling resistant)	* SiO <sub>2</sub> 4 Al <sub>2</sub> O <sub>3</sub> 2 Fe <sub>2</sub> O <sub>3</sub> 2 CaO 5 MgO 87	24.2	2.67	167	> 1730	7170	—	Failed after 11 minutes at 1600°C.	2 hours at 1500°C. 1.0 per cent. (Con.)
Chromemagnesite	* SiO <sub>2</sub> 4.6 Al <sub>2</sub> O <sub>3</sub> 13.4 Fe <sub>2</sub> O <sub>3</sub> 12.9 MgO 45.5 Cr <sub>2</sub> O <sub>3</sub> 20.9	21.2	3.00	188	> 1730	3660	Initial deformation 1430°C. Fail 1650°C.	* Failed after 9 minutes at 1600°C.	2 hours at 1500°C. 0.1 per cent. (Exp.)
Stable Dolomite	* SiO <sub>2</sub> 12.6 Al <sub>2</sub> O <sub>3</sub> 2.2 FeO 3.2 CaO 38.6 MgO 40.9	22.1	2.58	161	> 1730	> 8300	Initial deformation 1540°C. Fail 1680°C.	Failed after 32 minutes at 1600°C.	2 hours at 1500°C. Volume stable
Fused Alumina	Al <sub>2</sub> O <sub>3</sub> 96	26.1	2.90	181	> 1730	> 7380	Initial deformation 1340°C. Fail 1700°C.	—	2 hours at 1500°C. 0.3 per cent. (Con.)
Forsterite	SiO <sub>2</sub> 38.2 Al <sub>2</sub> O <sub>3</sub> 1.9 Fe <sub>2</sub> O <sub>3</sub> 7.3 MgO 51.9 Cr <sub>2</sub> O <sub>3</sub> 1.0	20.3	2.76	172	> 1730	3170	—	1600°C. for 1 hour 6.2 per cent. collapse	2 hours at 1500°C. 0.4 per cent. (Exp.)

\* Properties were obtained on bricks of similar type, but not on bricks taken from the same batch.

DIX 14—*continued*REFRACTORY BRICKS—*continued*

<i>Thermal shock resistance</i>	<i>Resistance to slags</i>	<i>Thermal expansion (20°–1000°C.)</i>	<i>Thermal conductivity</i>	<i>Specific heat</i>	<i>Comments</i>
Excellent	Excellent resistance to basic slags and particularly to iron oxide and cement clinker	*1.3%	*25 B.Th.U. —1000°C. mean	*0.29 <sub>5</sub>	Mainly employed in positions where resistance to strongly basic slags is essential, <i>e.g.</i> , under hearths and in linings of basic open-hearth furnaces. Used in hearths of reheating furnaces where mill scale makes the use of fireclay bricks undesirable.
Excellent	Excellent resistance to basic slags and iron oxide	*0.8%	*10 B.Th.U. —1000°C. mean	*0.25	Widely used in the construction of open-hearth furnaces (including the roof), in the hearths of reheating furnaces and soaking pits.
Low	High resistance to basic slags, but inferior in this respect to magnesite	*1.3%	*About 15 B.Th.U. —1000°C. mean	0.25 <sub>5</sub>	Gives good service under the hearth and in the banks of basic open-hearth furnaces. Also used in reheating furnace hearths.
Excellent	Good resistance to iron oxide and also to glass	0.6%	*High compared with fireclay brick	*0.27	Mainly employed in the construction of laboratory furnaces required to operate at temperatures up to 2000°C.
Moderate	Resistance to basic slags intermediate between that of chrome-magnesite and silica	*1.0%	*About 7 B.Th.U. —400°C. mean	*0.27 <sub>5</sub>	Gives good service in certain positions in open-hearth furnaces, <i>e.g.</i> , back walls. Used in cement kilns and copper furnaces.

APPEN  
PROPERTIES OF SELEC

<i>Brick type</i>	<i>Thermal conductivity</i>		<i>Maximum safe temperature</i>	<i>Cold crushing strength (lb./sq. in.)</i>	<i>Porosity (per cent.)</i>
	<i>Mean temp. °C.</i>	<i>B.Th.U.</i>			
<i>Low Temperature</i> <i>Diatomite Slabs</i> <i>(As mined)</i>	300	0.55	900°C.	240	74.3
	400	0.58			
	450	0.61			
<i>Low Temperature</i> <i>(Diatomite—solid grade)</i>	300	1.19	850°C.	410	66.3
	400	1.22			
	450	1.25			
<i>Low Temperature</i> <i>(Diatomite—light grade)</i>	400	0.84	850°C.	140	74.0
	450	0.88			
	500	0.92			
<i>Low Temperature</i> <i>(Asbestos base)</i>	300	0.64	900°C.	60	Not determined
	400	0.68			
	500	0.72			
<i>Intermediate Temperature</i> <i>(Diatomite base)</i>	350	1.13	1100°C.	980	60.2
	400	1.17			
	500	1.23			
<i>High Temperature</i> <i>(Porous fireclay)</i>	300	1.58	1400°C.	400	68.0
	400	1.66			
	500	1.78			

## DIX 15

## TED INSULATING BRICKS

<i>Bulk density</i>		<i>Permeability c.g.s. units</i>	<i>Comments</i>
<i>g. per ml.</i>	<i>lb. per cu. ft.</i>		
0.52	32.4	0.0006	An excellent low temperature insulating brick. It has a moderately good crushing strength, but shows a tendency to split along the laminations.
0.74	46.1	0.003	Contains a considerable proportion of clay bond which gives greater strength, but higher conductivity than the "light" diatomite brick. Used as a backing brick or exposed in low temperature stoves, etc.
0.55	34.1	0.180	A high grade insulating brick used where good insulation is essential, the temperature less than 850°C. and high cold crushing strength not required, e.g., on the outside of reheating furnaces.
0.54	33.4	0.004	Possesses, in addition to low conductivity, the ability to crush without crumbling. Hence used for insulation of blast furnace stoves and under open-hearth furnace bottoms, etc.
0.64	41.6	0.051	An excellent diatomite insulating brick for use in positions where the hot face temperature may reach 1100°C. Applications for such bricks would at present be limited.
0.80	49.6	1.3	Gives excellent service as high temperature backing brick, e.g., in the top of open-hearth checker chambers and as hot face insulation in forge and reheating furnaces, particularly if coated with suitable cement. Used extensively in the construction of reheating furnaces, recuperators, etc.

## APPENDIX 16

## MISCELLANEOUS SPECIAL REFRACTORIES

During recent years there has been a considerable increase in the number of refractory materials potentially useful to steelplant operators, though little change in the materials actually employed. Many of the new refractories, which include sulphides, carbides and nitrides, have been developed specifically for use in atomic energy or gas turbines, but few if any of these are likely to be used in steelworks other than for purely scientific purposes, e.g., thermocouple sheaths. Certain refractories, notably those described below may, however, well find an extended use in the steelplant.

1. BERYLLIUM OXIDE (*Beryllia*,  $\text{BeO}$ )

The properties of this refractory, which is prepared from the silicate mineral beryl ( $3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ) have been usefully described by Norton in 1947.\* For refractory purposes the oxide used has a purity of over 99 per cent. and a melting point of  $2510^\circ\text{C}$ . Its density is rather less than that of magnesia (3.0 compared with 3.58), whilst its hardness is high, Mohs' scale—9. Its thermal shock resistance is stated to be excellent, due doubtless in part to its high thermal conductivity. Unlike most other basic oxides it shows remarkable volatility at temperatures of  $1800^\circ\text{C}$ . and over. Norton referring to a loss in weight of 50 per cent. on crucibles fired for several hours at this temperature.

Several procedures are available for preparing beryllia ware, one being to use a high temperature calcined or fused grog, together with about one-third of its weight of low temperature calcined oxide. Such a mixture after blending with water and tamping in a mould with and without an organic binder merely requires firing to produce the finished ware. Where high density is required the firing temperature is usually  $1800^\circ\text{C}$ . or over, but the time at top temperature relatively short. So far the main use of beryllia is as a container for the melting of pure metals and alloys.

Incidentally mention should be made of the health hazard associated with the use of beryllia. Certain individuals show a high susceptibility to beryllia dust, contracting a serious lung condition from even small quantities, where other individuals work with it continually without serious trouble.

\**J. Amer. Ceram. Soc.*, 1947, 30, 242.

## 2. CARBON (C)

Very extensive use has been made of carbon bricks in the hearths, boshes, and even in the stacks of blast furnaces. Its use in most steelplant furnaces would, however, be subject to one serious difference in operating conditions, viz., the presence of free oxygen. Given a carbon brick that would not burn at high temperature in the presence of appreciable amounts of oxygen, there is little doubt that carbon would be widely used in the steelplant. Bricks made from coke-tar, or anthracite-tar, have remarkable properties, in particular extremely high refractoriness and refractoriness-under-load, excellent abrasion resistance, good thermal shock resistance and a relatively low thermal expansion (0.65 per cent. for  $20^\circ$  to  $1000^\circ\text{C}$ .).

Such a brick would be ideal, for example, for use in open-hearth furnace roofs if it were not for the readiness with which it oxidises. So great indeed are its potentialities that consideration has been given to the possibility of its use in conjunction with a protective atmosphere. In certain steelplant furnaces, for example the cupola, limited use is already being made of carbon, the difficulties of combustion being more than offset by its remarkable resistance to slag attack. This latter is incidentally well illustrated by the use of carbon crucibles in research work, *e.g.*, for melting blast furnace slags. Unfortunately it cannot be employed where reducible oxides, such as  $\text{FeO}$ , are present, since under these conditions the slags change in composition during melting.

The possibility of producing a carbon brick not subject to combustion has been considered by many workers. It has been shown that the rate of combustion can be markedly decreased, *e.g.*, by impregnation with various compounds, but the residual combustion tendencies are still too great to make their general use in steelplants at all practicable.

### 3. FORSTERITE ( $2\text{MgO} \cdot \text{SiO}_2$ )

The pure mineral forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ) has a melting point of over  $1900^\circ\text{C}$ . Forsterite refractories on the other hand are usually made either from natural olivines which contain fayalite ( $2\text{FeO} \cdot \text{SiO}_2$ ) in solid solution, or mixtures of serpentine ( $3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) with magnesia. Although the refractoriness of these commercial products is much less than that of the pure material, they are nevertheless very refractory, and in addition possess a high refractoriness-under-load.

Forsterite was first of interest in the steelplant because it constituted the bond in most well-fired chrome-magnesite bricks. It has, however, now achieved a place in its own right: forsterite bricks and forsterite-chrome bricks being used quite extensively in the top courses of checker fillings and in such positions as open-hearth furnace downtakes. Bricks of this type are frequently somewhat cheaper than chrome-magnesite bricks, but unfortunately have a rather lower slag resistance. Their thermal conductivity is similar to that of silica bricks, whilst the thermal expansion is about 1 per cent. for the temperature range  $20^\circ$  to  $1000^\circ\text{C}$ . A study of the patent literature shows that forsterite is still in an active state of development, a recent improvement, for example, being the addition of alumina to give improved stability in bricks used in open-hearth checkers.

### 4. SILICON CARBIDE ( $\text{SiC}$ )

Silicon carbide is not found in nature, except in meteorites, being a synthetic product formed by heating together coke, sand, salt and sawdust, in an electric resistance furnace. The salt volatilises various impurities in the coke and sand, whilst the sawdust gives a certain degree of porosity and enables the carbon monoxide formed by reaction between the coke and sand to escape.

Silicon carbide decomposes rather than melts, but can be used under certain conditions at temperatures of over  $2000^\circ\text{C}$ . The properties of bricks made from it vary greatly according to whether they are made from crushed silicon carbide with an added bond, *e.g.*, clay, or solely from silicon carbide refired at a very high temperature.

The use of silicon carbide in steelplants has so far been extremely limited, due in the main to its tendency to change its nature under combustion conditions. One of its main applications at present is in the form of tubes for recuperators, where its high conductivity and high thermal shock resistance are a great advantage. Precise data regarding its resistance to attack by iron-rich slags is singularly lacking, but it is known to stand up particularly well to slags of the coal ash type. But for its high initial cost it would doubtless find a much greater application in steelplants than it does at present.

#### 5. THORIA ( $ThO_2$ )

The oxide thoria is obtained by magnetic separation and chemical treatment from monazite sands, in which it is present as a complex phosphate. It is also found in certain black sand deposits associated with zircon and ilmenite. Although it has both an extremely high refractoriness (over  $2500^{\circ}C.$ ) and a good resistance to basic slags, its application in steelplants is likely to be severely limited by the high price per brick: the latter being due partly to scarcity but also to its extremely high specific gravity—9.5 to 10.

#### 6. ZIRCONIA ( $ZrO_2$ ) and ZIRCON ( $ZrO_2 \cdot SiO_2$ )

Zirconia occurs in nature as the mineral baddeleyite, which is found in Brazil and contains 80 to 90 per cent. of  $ZrO_2$ . Much more readily available is the silicate mineral zircon, with which zirconia is frequently confused. Zircon—whose theoretical composition is 67.2 per cent.  $ZrO_2$  and 32.8 per cent.  $SiO_2$ —is found in Brazil, U.S.A., Australia and India, generally in association with quartz, garnet, rutile, and other minerals.

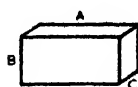
Zirconia, like thoria, has an extremely high melting point ( $2700^{\circ}$  to  $2900^{\circ}C.$ ) and a relatively high specific gravity—5.8. Its use has, however, been limited not only by its high price but by its unfortunate inversion on heating, which like that of silica leads to extreme sensitivity to thermal shock in certain regions. It has been shown, however, that zirconia can be stabilised by the addition of various oxides, notably 10 to 20 per cent. of lime. Ware made from this material does not show the normal sensitivity to thermal shock and is already finding application in the steelplant, *e.g.*, in the production of nozzles for continuous casting, where good thermal shock resistance and extremely high slag resistance are both essential. Zircon, which is considered by some workers to have appreciably less resistance to iron oxide, has also been successfully applied for this purpose. It has been shown that at high working temperatures (above  $1550^{\circ}C.$ ) it tends to dissociate into zirconia and silica glass.

Both zircon and zirconia have also been used as additions to other refractories, *e.g.*, to magnesite, in basic induction furnace linings. Here again the probability of either zircon or zirconia bricks being used in large quantities in steelplants would appear remote.

## APPENDIX 17

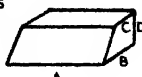
## NOMENCLATURE OF BRICK SHAPES

The more common types of brick shapes are summarised, both American and British names being given. The sizes illustrated are the 3 in. series, which is popular in Great Britain. In the U.S.A. the 2½ in. series is more commonly employed.



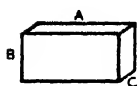
SQUARES OR 9'S STRAIGHTS

A B C  
9" x 4½" x 3"



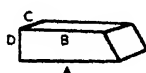
SIDE SKEW OR BEVEL SIDE.

A B/C D  
9" x 4½" x 3"



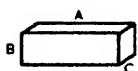
SPLITS OR SCONES

A B C  
9" x 4½" x 2"



END SKEW OR BEVEL END.

A/B C D  
9/7" x 4½" x 3"



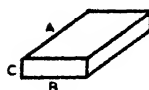
SOAPS PUPS OR CLOSERS.

A B C  
9" x 3" x 2¼"



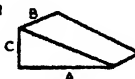
FEATHER SIDE OR FEATHER EDGE.

A B C  
9" x 4½" x 3/0"



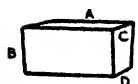
BEARERS BLOCKS OR QUARRIES.

A B C  
16" x 9" x 3"



FEATHER END OR NECK BRICK.

A B C  
9" x 4½" x 3/0"



SIDE ARCH OR ARCH.

A B C/D  
9" x 4½" x 3/2"



BULLNOSE OR JAMB BRICK.

A B C  
9" x 4½" x 3-½" RAD.



END ARCH OR WEDGE.

A B C/D  
9" x 4½" x 3/2"



CIRCLE OR CUPOLA BRICKS.

A/B C D  
9/6" x 4½" x 3"



CUPOLA. KEY, CROWN OR BULLHEAD.

A B/C D  
9" x 4½/24" x 3"



# APPENDIX 18 NUMBER OF BRICKS REQUIRED TO TURN VARIOUS CIRCLES

The quantities shown are for complete rings in each case

Inside Diameter of Circle	9-inch End Arch Bricks				4½-inch Side Arch Bricks				9-inch Cupola, Crown, Key, or Bullhead Bricks				4½-inch Circle, or Cupola Lining Bricks		
	3'		2½"		3'		2½"		3'		2½"		Taper		Quantity
	Taper	Quantity	Taper	Quantity	Taper	Quantity	Taper	Quantity	Taper	Quantity	Taper	Quantity	Inches		
Ft. ins.	Inches		Inches		Inches		Inches		Inches		Inches		Inches		
1 0 ..	3/1 1/8	32	2 1/4	38	3/1 1/8	22	2 1/4	26	4 1/4	21	9/5 1/8	7 1/2			
1 6 ..	3/1 1/8	38	2 1/4	45	3/2	28	2 1/4	35	4 1/4	26	9/6	9 1/2			
2 0 ..	3/1 1/8	44	2 1/4	55	3/2 1/8	34	2 1/4	40	4 1/4	30	9/6 1/8	11 1/2			
2 6 ..	3/1 1/8	50	2 1/4	60	3/2 1/8	41	2 1/4	49	4 1/4	34	9/6 1/8	13 1/2			
3 0 ..	3/2	57	2 1/4	64	3/2 1/8	48	2 1/2	57	4 1/4	38	9/7 1/8	15 1/2			
3 6 ..	3/2 1/8	62	2 1/4	73	3/2 1/8	54	2 1/2	62	4 1/4	43	9/7 1/8	18			
4 0 ..	3/2 1/8	70	2 1/4	82	3/2 1/8	60	2 1/2	71	4 1/4	47	9/7 1/8	20 1/2			
4 6 ..	3/2 1/8	79	2 1/4	92	3/2 1/8	66	2 1/2	80	4 1/4	51	9/7 1/8	22 1/2			
5 0 ..	3/2 1/8	83	2 1/4	100	3/2 1/8	72	2 1/2	88	4 1/4	55	9/7 1/8	24			
5 6 ..	3/2 1/8	88	2 1/4	106	3/2 1/8	78	2 1/2	95	4 1/4	60	9/7 1/8	26 1/2			
6 0 ..	3/2 1/8	94	2 1/4	114	3/2 1/8	85	2 1/2	102	4 1/4	64	9/8	28 1/2			
7 0 ..	3/2 1/8	107	2 1/4	130	3/2 1/8	98	2 1/2	117	4 1/4	72	9/8 1/8	32 1/2			
8 0 ..	3/2 1/8	120	2 1/4	144	3/2 1/8	110	2 1/2	131	4 1/4	81	9/8 1/8	37			
9 0 ..	3/2 1/8	133	2 1/4	160	3/2 1/8	122	2 1/2	147	4 1/4	90	9/8 1/8	41			
10 0 ..	3/2 1/8	145	2 1/4	175	3/2 1/8	134	2 1/2	162	4 1/4	98	9/8 1/8	45			
11 0 ..	3/2 1/8	158	2 1/4	189	3/2 1/8	148	2 1/2	178	4 1/4	107	9/8 1/8	49 1/2			
12 0 ..	3/2 1/8	170	2 1/4	202	3/2 1/8	160	2 1/2	193	4 1/4	115	9/8 1/8	53 1/2			
13 0 ..	3/2 1/8	182	2 1/4	218	3/2 1/8	172	2 1/2	207	4 1/4	121	9/8 1/8	57			

Brick sizes given to the nearest 1/8th of an inch.

(General Refractories Ltd.)

**APPENDIX 18—continued**  
**NUMBER OF BRICKS REQUIRED TO TURN VARIOUS CIRCLES—continued**  
 The quantities shown are for complete rings in each case

Inside Diameter of Circle	9-inch End Arch Bricks			4½-inch Side Arch Bricks			9-inch Capola, Crown, Key, or Bullhead Bricks			4½-inch Capola, or Capola Lining Bricks		
	3'		2½"	3"		2½"	3'		2½"	Taper		Quantity
	Taper	Quantity		Taper	Quantity		Taper	Quantity		Inches	Quantity	
Ft. ins.	Inches		Inches	Inches		Inches	Inches		Inches	Inches		Inches
14 0 ..	3/2 11	194	2½/2 1	3/2 7	185	2½/2 1	2½/2 1	222	4½/4 11	9/8 11	129	9/8 11
15 0 ..	3/2 1	207	2½/2 1	3/2 7	197	2½/2 1	2½/2 1	237	4½/4 1	9/8 11	138	9/8 11
16 0 ..	3/2 1	219	2½/2 1	3/2 1	210	2½/2 1	2½/2 1	252	4½/4 1	9/8 11	146	9/8 11
17 0 ..	3/2 1	232	2½/2 1	3/2 1	223	2½/2 1	2½/2 1	267	4½/4 1	9/8 11	155	9/8 11
18 0 ..	3/2 1	245	2½/2 1	3/2 1	235	2½/2 1	2½/2 1	282	4½/4 1	9/8 11	163	9/8 11
19 0 ..	3/2 1	257	2½/2 1	3/2 1	248	2½/2 1	2½/2 1	297	4½/4 1	9/8 11	171	9/8 11
20 0 ..	3/2 1	270	2½/2 1	3/2 1	260	2½/2 1	2½/2 1	312	4½/4 1	9/8 11	180	9/8 11
21 0 ..	3/2 1	282	2½/2 1	3/2 1	273	2½/2 1	2½/2 1	327	4½/4 1	9/8 11	188	9/8 11
22 0 ..	3/2 1	295	2½/2 1	3/2 1	285	2½/2 1	2½/2 1	343	4½/4 1	9/8 11	196	9/8 11
23 0 ..	3/2 1	308	2½/2 1	3/2 1	298	2½/2 1	2½/2 1	358	4½/4 1	9/8 11	205	9/8 11
24 0 ..	3/2 1	320	2½/2 1	3/2 1	311	2½/2 1	2½/2 1	373	4½/4 1	9/8 11	213	9/8 11
26 0 ..	3/2 1	345	2½/2 1	3/2 1	336	2½/2 1	2½/2 1	403	4½/4 1	9/8 11	230	9/8 11
28 0 ..	3/2 1	370	2½/2 1	3/2 1	361	2½/2 1	2½/2 1	433	4½/4 1	9/8 11	247	9/8 11
30 0 ..	3/2 1	396	2½/2 1	3/2 1	386	2½/2 1	2½/2 1	463	4½/4 1	9/8 11	264	9/8 11
32 0 ..	3/2 1	421	2½/2 1	3/2 1	411	2½/2 1	2½/2 1	493	4½/4 1	9/8 11	280	9/8 11
34 0 ..	3/2 1	446	2½/2 1	3/2 1	436	2½/2 1	2½/2 1	524	4½/4 1	9/8 11	297	9/8 11
36 0 ..	3/2 1	471	2½/2 1	3/2 1	462	2½/2 1	2½/2 1	554	4½/4 1	9/8 11	314	9/8 11

Brick sizes given to the nearest 1/8th of an inch.

(General Refractories Ltd.)

## APPENDIX 19

## USEFUL UNITS AND CONVERSION FACTORS

## MASS, LENGTH, AREA, AND VOLUME

Angström unit (Å or Å.U.)— $3.937 \times 10^{-8}$  inch;  $1 \times 10^{-10}$  metre;  $1 \times 10^{-8}$  centimetre.

Centimetre (cm.)—0.39370 inch; 0.01 metre; 10 millimetres.

Cubic centimetre (cm<sup>3</sup>)—0.061023 cubic inch.

Cubic foot (ft<sup>3</sup> or cu. ft.)—1728 cubic inches;  $2.8317 \times 10^4$  cubic centimetres.

Cubic inch (in<sup>3</sup> or cu. in.)— $5.787 \times 10^{-4}$  cubic foot; 16.387 cubic centimetres.

Cubic metre—35.31 cubic foot.

Foot (ft.)—30.48 centimetres.

Gallon (British)—4.546 litres (1 British gallon of water at 15°C. weighs 10 lb.).

Gallon (U.S.)—3.785 litres (1 U.S. gallon of water at 15°C. weighs 8.337 lb.).

Gram (g.)—0.0022 pounds (Av.); 0.0353 ounces (Av.).

Hundredweight (cwt.) (short)—100 pounds; 45.3592 kilograms.

Hundredweight (cwt.) (long)—112 pounds; 50.8023 kilograms.

Inch (in.)—2.540 centimetres.

Kilogram (kg.)—2.2046 pound (Av.).

Litre (l.)—0.0353 cubic feet; 61.025 in.<sup>3</sup>

Metre (m.)—39.370 inches.

Micron—0.001 millimetres.

Ounce (avoirdupois) (oz.)—28.349 grams.

Pound (avoirdupois) (lb.)—453.59 grams.

Square centimetre (cm<sup>2</sup>)—0.155 square inches.

Square foot (ft<sup>2</sup>)—0.0929 square metres.

Square inch (in<sup>2</sup>)—6.4516 square centimetres.

Square yard—0.836 square metres.

Square metre (m<sup>2</sup>)—10.76 square feet

Ton (long)—2240 pounds.

Ton (short)—2000 pounds.

Tonne (metric ton)—2204.62 pounds.

## DENSITY

Grams per cubic centimetre—0.03613 pounds per in<sup>3</sup>; 62.43 lb. per ft<sup>3</sup>;  
1.000028 g.p.ml.

Pounds per cubic foot—0.0160 grams per cm<sup>3</sup>.

Pounds per cubic inch—27.68 grams per cm<sup>3</sup>.

## PRESSURE

Atmosphere (normal)—14.695 pounds per square inch; 29.92 in. or 760 mm.  
Hg; 1.033 kg. per cm<sup>2</sup>.

Grams per square centimetre—0.0142 pound per square inch; 2.0482  
pounds per square foot.

Kilograms per square centimetre—14.223 pounds per square inch.

Pounds per square foot—4.8824 kilograms per square metre.

Pounds per square inch—70.307 grams per square centimetre.

## POWER, WORK, AND THERMAL UNITS

British Thermal Unit (mean) (B.Th.U.)— $2.930 \times 10^{-4}$  kilowatt-hour;  
251.98 gram calories.

Centigrade Heat Unit (C.H.U.)—1.8 B.Th.U.

Erg— $2.3889 \times 10^{-8}$  gram calories;  $7.3756 \times 10^{-8}$  foot pounds.

Foot pound— $3.7662 \times 10^{-7}$  kilowatt-hour; 0.32389 gram calories.

Gram calorie (mean)—0.0039685 B.Th.U. (mean).

Horse power—746 watts.

Kilogram calorie or large calorie (mean)—3.9685 B.Th.U. (mean) = 1000 g.cal.

Therm—100,000 B.Th.U.

Watt—0.001 kilowatt; 0.00134 horse power; 3.413 B.Th.U./hr.

## TEMPERATURE

Degree centigrade— $\frac{9}{5}$  Fahrenheit.

Degree Fahrenheit— $\frac{5}{9}$  centigrade.

To convert centigrade to Fahrenheit:

$$C^{\circ} = \frac{5}{9} (F^{\circ} - 32^{\circ})$$

$$F^{\circ} = \frac{9}{5} C^{\circ} + 32^{\circ}$$

## THERMAL CONDUCTIVITY

*Gram calories* per square centimetre per second for a temperature gradient of  $1^{\circ}\text{C}$ . per centimetre = 2902.32 British Thermal Units per square foot per hour for a temperature gradient of  $1^{\circ}\text{F}$ . per inch.

## APPENDIX 20\*

## METHODS OF TESTING REFRACTORY MATERIALS

BRITISH STANDARD 1902 : 1952

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## BRITISH STANDARD METHODS OF TESTING REFRACTORY MATERIALS

### FOREWORD

The testing of refractory materials to assess their quality and uniformity has been practised in many branches of industry throughout the past 40 years. The experience thus gained has resulted in a measure of standardisation of techniques, and the first corporate body in Great Britain to issue a description of standard testing methods was the Institution of Gas Engineers. Much of the work on which their standard methods were based was carried out under the direction of the late Dr. J. W. Mellor, first in the laboratories of the North Staffordshire Technical College and subsequently in the laboratories of the British Refractories Research Association (now incorporated in the British Ceramic Research Association). With this broad foundation of experience, the Testing Committee of the Research Association began its work in 1938, and has since carried out a considerable number of co-operative experimental investigations.

In 1941, the Testing Committee issued a first draft of a series of tentative methods of testing refractories of the fireclay, silica and siliceous types. On the basis of the comments received from the manufacturing and using industries and in the light of further experimental work carried out by the Committee, the draft was revised and re-issued in 1946; this revised draft has been used as a basis for the present British Standard.

This British Standard is confined to methods of testing having a general field of application, and it forms the first part of what is ultimately to be a more comprehensive publication. The standard relates more particularly to fireclay and silica refractories, but work is in hand on which it is hoped to base British Standard methods of testing basic refractories, insulating refractories, refractory cements and patching materials.

Work is also in preparation on the measurement of warpage and size, and it is hoped that methods will be available for inclusion in the first revision of this British Standard.

The testing of refractory materials presents many difficulties and the work should only be carried out by those having the necessary experience in the use and maintenance of the apparatus employed.

### SPECIFICATION

#### PART I. GENERAL PRINCIPLES AND METHODS OF SAMPLING

##### *Section 1: The Classification of Refractory Materials*

#### CLASSIFICATION AND DEFINITIONS

1. The classification of refractory materials and their definitions for the purposes of this British Standard are as follows:

(a) *Silica brick*. A brick that, in the fired state, shows on analysis 92 per cent. or more of silica.

(b) *Siliceous brick*. A brick that, in the fired state, shows on analysis at least 78 per cent. but less than 92 per cent. of silica, the remainder being

essentially alumina. (Refractories known as "semi-silica bricks" and "semi-siliceous firebricks" fall within this group.)

(c) *Firebrick*. A brick that, in the fired state, consists essentially of aluminosilicates and silica, and shows on analysis less than 78 per cent. of silica and less than 38 per cent of alumina.

(d) *Aluminous firebrick*. A brick that, in the fired state, shows on analysis from 38 to 45 per cent. of alumina, the remainder being essentially silica.

(e) *Other refractory materials, cements, etc.* Those types of refractory not included in the preceding classification are usually described under generic terms which are sufficiently definite for the present needs of industry; such terms are magnesite brick, chrome brick, chrome-magnesite brick, sillimanite brick, silicon carbide brick, etc.

### *Section 2: Sampling*

#### INTRODUCTION

2. The importance of careful sampling as a basis for routine testing has now become widely recognised, and many manufacturing processes in which articles are made to standard specifications are controlled in this way. Regular sampling, however, is more readily achieved with some manufactured articles than with others; the problem is simplified, for example, with a homogeneous material. The refractories and heavy clay industries, on the other hand, are based on comparatively heterogeneous raw materials, which become more complex at each stage of the manufacturing process. For this reason, a technique for systematic sampling in the refractories and heavy clay industries has not yet been prepared. Indeed it seems unlikely that a rigid system of sampling can be devised that will have general application to the clay industries. It is the purpose of this section, therefore, to indicate only general principles. With increasing experience it may become possible to give these fuller definition.

#### GENERAL

3. A sample is defined as a portion of material or a group of units, taken from a large mass of aggregation which is used to give information on the quality of the larger quantity.

Samples may be classified as "random" or "representative". Random samples are taken without bias so that every item has an equal chance of inclusion. Representative samples are drawn in a systematic way so that each portion of the sample represents a corresponding portion of the bulk. The sampling of each portion will probably need to be random.

Each of these methods will be used as appropriate in the sampling of refractory products. Representative samples may be taken by the manufacturer from a kiln to check the uniformity of firing, and random samples may be taken by the user from a consignment of bricks to check the quality of the material.

Proper sampling can only be done when the whole of the batch from which the sample is required is accessible to the sampler. In general this is only accomplished when the material is being moved from one position to another. A stock pile cannot adequately be sampled under practical conditions.

**SIZE OF SAMPLE**

4. The size of the sample is determined by many factors, among which are the statistical uniformity of the material, the accuracy required in the result of the tests, the nature of the tests that are to be made and, sometimes, the number of units in the batch. It is usually found that the first factor necessitates a much larger sample when certain properties, such as size, are being investigated than when others, such as refractoriness, are under consideration. The limitations of the testing equipment available will frequently determine the maximum number of units that can be sampled and tested. The last factor applies particularly to the sampling of shapes, which present special problems, among which are the difficulty in maintaining regularity in the withdrawal of increments from mixed batches and the necessity of having a large ratio of increments to bulk for the adequate sampling of small batches.

Although, statistically, the size of the sample required is not dependent on the size of the batch being sampled, it is usually convenient to base a sampling schedule to some extent on the number of units being sampled; the following is accordingly offered for guidance in the formulation of a sampling programme:

*Stock size bricks.* For the complete series of tests a minimum sample should consist of 10 bricks.

For batches of more than 30,000 bricks, the minimum rate of sampling should be 1 brick per 3000.

*Special shapes and sizes.* For the reasons outlined above, no general rules can be given, but it is suggested that, whenever convenient, the procedure for stock size bricks should be followed.

*Ground clay and other ground materials, cements, etc.* The following sampling recommendations apply only to materials below 0.5 in. in maximum grain size:

A minimum representative sample of 10 lb. should be taken.

For batches of more than 5 tons, the rate of sampling should be not less than 2 lb. per ton.

**QUALITY CONTROL**

5. Since one of the objects of testing is insurance against quality fluctuations, attention is directed to the advantages of the quality control chart. This chart has been aptly described as a graphical record of quality, and it can be usefully adapted to a manufacturing process such as brickmaking. Its wider use in any continuous scheme of sampling and testing is to be commended. For a concise statement of the technique involved, reference should be made to B.S. 600R, "Quality control charts".

**PART II. STANDARD METHODS OF CHEMICAL ANALYSIS****STANDARD TEST NO. 1****CHEMICAL ANALYSIS OF SILICA AND SILICEOUS  
REFRACTORIES, FIREBRICKS AND ALUMINOUS FIREBRICKS***Section 1: Sampling*



## BRICKS

6. Take at least 3 bricks which should be representative of the ten-brick sample (see Clause 4 of Section 2, Part 1). Break up a half of each brick, including any skin\* in the sample, and crush to pass a No. 7 mesh B.S. test sieve.† Mix, cone and quarter to obtain about 200 g. of sample and crush this to pass a No. 30 mesh B.S. test sieve. For samples that are likely to contain magnetic material, determine the iron in a separate sample ground in a Wedgwood mortar. Mix, cone and quarter to obtain about 20 g. of sample, and crush in a hardened steel percussion mortar to pass a No. 120 mesh B.S. test sieve, removing any particles of extraneous iron with a magnet. The resulting material is suitable for the determination of hygroscopic water. Obtain the final sample for analysis by finely grinding portions as required in an agate mortar.

## GROUND MATERIALS

7. Cone and quarter the 10 lb. sample (see Clause 4 of Section 2, Part 1) to obtain about 2 lb. of material and crush, if necessary, to pass a No. 7 mesh B.S. test sieve. Mix, cone and quarter to obtain about 200 g. of sample and crush to pass a No. 30 mesh B.S. test sieve as with brick samples. For samples that are likely to contain magnetic material determine the iron in a separate sample ground in a Wedgwood mortar. Mix, cone and quarter to obtain about 20 g. of sample and crush in a hardened steel percussion mortar to pass a No. 120 mesh B.S. test sieve, removing any particles of extraneous iron with a magnet. The resulting material is suitable for the determination of hygroscopic water. Obtain the final sample for analysis by finely grinding portions as required in an agate mortar.

*Section 2: Reagents Required*

## REAGENTS

8. All reagents shall be of analytical quality and of guaranteed purity and distilled water shall be used throughout the analysis.

## SOLUTIONS

9. *Alcohol mixture A.* Mix 97 ml. of absolute alcohol, 3 ml. of water and 0.25 ml. of perchloric acid (sp. gr. 1.20). Saturate with potassium perchlorate.

*Alcohol mixture B.* Mix 50 ml. of absolute alcohol with 50 ml. of anhydrous ether.

*Alcohol saturated with sodium magnesium uranyl acetate.* Precipitate sodium magnesium uranyl acetate from sodium chloride solution and shake the precipitate with absolute alcohol. Set the suspension aside and filter immediately before use.

*Ammonia solution.* Sp. gr. 0.880.

*Ammonia solution (1 : 1).* Dilute 500 ml. of ammonia solution (sp. gr. 0.880) to 1 litre with water.

*Ammonia solution (1 : 39).* Dilute 10 ml. of ammonia solution (sp. gr. 0.880) to 400 ml. with water.

\* Any fuel ash should be excluded as its presence will give misleading results.

† B.S.410, "Test sieves".

*Ammonium carbonate* (20 per cent.). Add 100 ml. of ammonia solution (sp. gr. 0.880) to 100 g. of ammonium carbonate, and dilute to 500 ml. with water. Stir to dissolve.

*Ammonium nitrate* (1 per cent.). Dilute 10 ml. of nitric acid (sp. gr. 1.42) to about 200 ml. with water and neutralise with ammonia solution (1 : 1). Dilute to one litre with water and render the solution faintly alkaline to methyl red.

*Ammonium oxalate* (1 per cent.). Dissolve 10 g. of ammonium oxalate  $(\text{COONH}_4)_2\text{H}_2\text{O}$ , in water and dilute to one litre.

*Ammonium oxalate* (4 per cent.). Dissolve 4 g. of ammonium oxalate  $(\text{COONH}_4)_2\text{H}_2\text{O}$ , in water and dilute to 100 ml.

*Ammonium phosphate* (10 per cent.). This solution should be freshly prepared. Dissolve 2 g. of diammonium hydrogen phosphate in water and dilute to 20 ml.

*Barium chloride* (10 per cent.). Dissolve 10 g. of barium chloride,  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ , in water and dilute to 100 ml.

*Bromine water* (saturated). Shake 500 ml. of water with about 20 ml. of bromine in a glass-stoppered bottle.

*Hydrochloric acid*. Sp. gr. 1.16.

*Hydrochloric acid* (1 : 1). Dilute 500 ml. of hydrochloric acid (sp. gr. 1.16) to one litre with water.

*Hydrochloric acid* (1 : 99). Dilute 10 ml. of hydrochloric acid (sp. gr. 1.16) to one litre with water.

*Hydrofluoric acid*. 40 per cent. w/w.

*Hydrogen peroxide* (20 vol.).

*Magnesium uranyl acetate* (saturated). *Solution A*. Add 57 ml. of glacial acetic acid and one litre of water to 85 g. of crystallised uranyl acetate  $(\text{CH}_3\text{COO})_2\text{UO}_2 \cdot 2\text{H}_2\text{O}$ .

*Solution B*. Add 57 ml. of glacial acetic acid and one litre of water to 500 g. of crystallised magnesium acetate  $(\text{CH}_3\text{COO})_2\text{Mg} \cdot 6\text{H}_2\text{O}$ .

Heat both mixtures to about 70°C. and stir until the solids are dissolved. Mix solutions A and B and allow to stand for 12 hours for the excess magnesium uranyl acetate to separate. It is essential that the solution is saturated at the temperature at which the determination is to be carried out, e.g.,  $20 \pm 1^\circ\text{C}$ .

*Methyl red indicator*. Dissolve 0.1 g. of methyl red in 60 ml. of absolute alcohol and dilute to 100 ml. with water.

*Nitric acid*. Sp. gr. 1.42.

*Nitric acid* (1 : 1). Dilute 500 ml. of nitric acid (sp. gr. 1.42) to one litre with water.

*Perchloric acid*. Sp. gr. 1.20.

*Sulphide wash liquid* (acid). Add 20 ml. of sulphuric acid (sp. gr. 1.84) to one litre of water and pass a rapid stream of hydrogen sulphide gas through the solution for 10 minutes.

*Sulphuric acid*. Sp. gr. 1.84.

*Sulphuric acid* (1 : 1). Cautiously add 500 ml. of sulphuric acid (sp. gr. 1.84) to 500 ml. of water, cooling the solution and keeping it well mixed during the addition of the acid. Dilute to one litre with water.

*Sulphuric acid* (1 : 9). Cautiously add 100 ml. of sulphuric acid (sp. gr. 1.84) to 900 ml. of water, cooling the solution and keeping it well mixed during the addition of the acid. Dilute to one litre with water.

*Tartaric acid* (10 per cent.). Dissolve 10 g. of tartaric acid in water and dilute to 100 ml.

*Thiaglycollic acid*. 90 per cent. w/w.

#### STANDARD SOLUTIONS

10. *Iron solution (stock)*. Dissolve 1.2078 g. of ammonium ferric alum  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{Fe}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$ , in warm water and when cool add 5 ml. of sulphuric acid (sp. gr. 1.84). Cool and dilute to one litre with water.

*Iron solution (standard)*. Dilute 25 ml. of the stock iron solution to one litre with water. 1 ml. of the diluted solution contains 0.005 mg.  $\text{Fe}_2\text{O}_3$ .

*Potassium permanganate*. Dissolve about 0.8 g. of potassium permanganate in 250 ml. of water to obtain an approximately 0.1*N* solution; boil for 5 minutes, filter through a sintered glass crucible and when cold dilute to 250 ml. Standardise with sodium oxalate and dilute portions as required with water to obtain exactly 0.02*N* permanganate solution. Keep in an amber-glass bottle.

*Titanium solution (stock)*. Evaporate potassium titanyl oxalate,  $\text{K}_2\text{TiO}(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ , almost to dryness with sulphuric acid (sp. gr. 1.84). Extract the residue with water and boil to hydrolyse the titanium sulphate. Filter, wash the residue with water until it is free from sulphates, then ignite to  $\text{TiO}_2$ . Fuse 1 g. of the pure  $\text{TiO}_2$  with 10 g. of potassium pyrosulphate and dissolve in 200 ml. of water containing 20 ml. of sulphuric acid (sp. gr. 1.84). Cool and dilute to one litre with water.

*Titanium solution (standard)*. Dilute 10 ml. of the titanium stock solution to 100 ml. with water as required. 1 ml. of the diluted solution contains 0.1 mg.  $\text{TiO}_2$ .

#### "BLANK" DETERMINATIONS

11. A "blank" determination shall be carried out on all reagents in accordance with the general scheme of the analysis.

#### Section 3: Glassware

12. Beakers shall be of borosilicate glass. Graduated glassware shall comply with the requirements of:

B.S. 612 Nessler cylinders.

B.S. 700 Graduated pipettes and straight pipettes.

B.S. 846 Burettes and bulb burettes.

B.S. 1792 One-mark graduated flasks.

Sintered filters shall comply with B.S. 1752, "Sintered disk filters for laboratory use".

#### Section 4: Procedure

##### DETERMINATION OF HYGROSCOPIC WATER

13. Heat 5–10 g. of the air-dried sample (passing a No. 120 mesh B.S. test sieve) in an air oven at 110°C. to constant weight. The sample should be spread in a thin layer. Three to 4 hours' drying is usually sufficient. The determination of hygroscopic water shall be commenced at the same time as the samples are weighed for the main analysis and the alkali determinations.

## DETERMINATION OF LOSS ON IGNITION

14. Ignite 1 g. of the air-dried finely ground sample in a platinum crucible at  $1000 \pm 25^\circ\text{C}$ . to constant weight. Air should have free access to the sample to maintain oxidising conditions. Thirty minutes' ignition is usually sufficient. Record the loss of weight separately from the main analysis as loss on ignition.

The significance of the loss on ignition determination may vary according to the nature of the sample. The figure obtained may include a loss due to the oxidation of any carbonaceous matter and a gain due to the oxidation of any ferrous iron present.

## DETERMINATION OF SILICA, FERRIC OXIDE, TITANIA, ALUMINA, LIME AND MAGNESIA

15. (a) *Decomposition of the sample.* Fuse 1 g. of the air-dried, finely ground sample with about 7 g. of anhydrous sodium carbonate in a covered platinum crucible. Raise the temperature slowly until frothing ceases, then complete the fusion at  $1000^\circ\text{C}$ . for 30 minutes, occasionally swirling the melt to ensure thorough mixing. Quench the melt by immersing the bottom half of the hot crucible in cold water, then place the crucible and lid in about 100 ml. of hot water contained in a platinum or porcelain evaporating basin. Cover the basin with a clock-glass and add a few drops of absolute alcohol. Then gradually add 30 ml. of hydrochloric acid (sp. gr. 1.16). Warm until the melt is completely disintegrated and remove the crucible and lid, washing them thoroughly and scrubbing them with a rubber-tipped rod. Crush any lumps remaining in the solution.

(b) *Determination of silica.* Evaporate the solution obtained from the fusion to dryness, breaking up from time to time the crust that forms and hinders evaporation. When the residue is completely dry cover the basin with a clock-glass and drench the residue with about 20 ml. of hydrochloric acid (sp. gr. 1.16). Allow to stand for a few minutes, then add about 75 ml. of hot water to dissolve the salts. Digest on a steam bath for 5 minutes, then filter through a No. 41 Whatman paper (or equivalent). Transfer the silica to the filter with a jet of hot water; it is unnecessary to scrub the basin. Wash the residue 5 times with hot diluted hydrochloric acid (1 : 99) followed by hot water until it is free from chlorides. Reserve the residue and paper for the subsequent ignition, and transfer the filtrate and washings back to the evaporating basin. Again evaporate completely to dryness, cover the basin with a clock-glass and bake in an air oven for 1 hour at  $110^\circ\text{C}$ . Allow to cool, then drench the residue with about 20 ml. of hydrochloric acid (sp. gr. 1.16). Allow to stand for a few minutes, add about 75 ml. of hot water and digest for 5 minutes on a steam bath. Filter through a No. 40 Whatman paper (or equivalent) transferring the residue to the filter with a jet of hot water and scrubbing the basin with a rubber-tipped rod. Wash 5 times with hot diluted hydrochloric acid (1 : 99), followed by hot water until the residue is free from chlorides. Reserve the filtrate and washings for the determination of ferric oxide, titania and alumina.

Place the 2 residues and papers, without drying, in a weighed platinum crucible and heat cautiously to dry the residue and char the papers. Then burn off the carbon and finally ignite at  $1200^\circ\text{C}$ . for 30 minutes and then to constant weight. Allow to cool and weigh to obtain the weight of impure

silica. Moisten the weighed residue with a few drops of dilute sulphuric acid (1 : 1) and add about 10 ml. of hydrofluoric acid. Evaporate slowly to dryness on a sand-bath (or under a suitable radiant heater) in a fume-hood. Ignite the dry residue to 1200°C. for 5 minutes, allow the crucible to cool, and weigh. Subtract the weight of this residue from the weight of impure silica to obtain the amount of silica in the sample taken. If the residue weighs more than 5 mg. repeat the treatment with hydrofluoric and sulphuric acids to ensure that all the silica is removed.

Fuse the residue with a small quantity of sodium carbonate and dissolve in diluted hydrochloric acid (1 : 1). Add the solution to that reserved for the determination of ferric oxide, titania and alumina.

(c) *Determination of ferric oxide, titania and alumina content.* For accurate work dissolved platinum should be removed from the combined solutions containing the ferric oxide, titania and alumina. Precipitate the platinum by passing hydrogen sulphide through the solution and filter. Boil the filtrate to expel hydrogen sulphide and re-oxidise the iron by adding bromine water. Boil off the excess bromine.

To the solution (approx. 300 ml.) add 2 to 3 g. of solid ammonium chloride, warm the solution to about 80°C. and add diluted ammonia solution (1 : 1) with stirring until precipitation appears to be complete. Render the solution just alkaline to methyl red. Boil the alkaline solution for 2 minutes, allow to stand for 5 minutes for the precipitate to settle and filter through a No. 41 Whatman paper (or equivalent). Transfer the precipitate to the filter and wash 5 times with hot faintly ammoniacal ammonium nitrate solution (1 per cent.). Reserve the filtrate and washings and transfer the precipitate back to the precipitation beaker. Moisten the paper with hot diluted hydrochloric acid (1 : 1) and wash thoroughly with water, adding these washings to the precipitation beaker. Reserve the paper for ignition. Dissolve the residue in a slight excess of hydrochloric acid, add 1 g. of solid ammonium chloride and dilute the solution to about 300 ml. with water. Repeat the precipitation as before and when precipitation is complete add some macerated filter-paper pulp, or filter accelerator, and stir vigorously. Filter through a No. 41 Whatman paper (or equivalent) and wash the precipitate free from chlorides with hot faintly ammoniacal ammonium nitrate solution (1 per cent.). Add the filtrate and washings to the reserved solution and keep the precipitate and paper for ignition.

Acidify the combined reserved filtrates with hydrochloric acid and evaporate to about 150 ml. Make just alkaline to methyl red with diluted ammonia solution (1 : 1). Any precipitate should be filtered off, reprecipitated, washed as before and reserved for ignition. Acidify the solution, or the filtrate and washings if a precipitate is obtained, with hydrochloric acid and reserve for the determination of lime and magnesia.

Place the precipitates and papers reserved for ignition in a weighed platinum crucible and heat, slowly at first, to dry the precipitates and char the papers. Burn off the carbon and finally ignite the residue at 1200°C. to constant weight. The weight of mixed iron, titanium and aluminium oxides is thus obtained. Carefully fuse the weighed ignited oxides, in the same platinum crucible, with about 6 g. of iron-free potassium pyrosulphate. Cool the crucible and extract the melt in a beaker with about 150 ml. of water and 5 ml. of sulphuric acid (sp. gr. 1.84). For accurate work recover any

dissolved silica by evaporating the solution to fuming, then dilute with water and filter. Ignite the residue and treat with hydrofluoric and sulphuric acids to obtain the weight of recovered silica. Add the weight of this silica to the weight of silica already obtained and make a corresponding deduction from the weight of mixed iron, titanium and aluminium oxides.

Dilute the solution of the pyrosulphate melt, or the filtrate from the silica recovery, to exactly 250 ml. in a graduated flask. The solution thus obtained will be referred to as the "stock solution".

(i) *Determination of ferric oxide.* Determine the iron colorimetrically by the thioglycollic acid method if 1 per cent. or less of ferris oxide is present, or volumetrically if more than 1 per cent. of ferric oxide content is present.

NOTE: Instead of the given procedure with Nessler cylinders for the colorimetric method, suitable colorimeters or absorptimeters may be used. These instruments generally give a greater accuracy and range than do visual determinations.

*Thioglycollic acid method.* Dilute 10 ml. of the "stock solution" to exactly 250 ml. with water in a graduated flask to obtain the "test solution". Fill a burette with standard iron solution (1 ml. = 0.005 mg.  $\text{Fe}_2\text{O}_3$ ) and another with distilled water. Use a pair of matched 100 ml. Nessler cylinders. Add 10 ml. of water to 10 ml. of tartaric acid solution (10 per cent.) in Nessler cylinder (a) and add 10 ml. of "test solution" to 10 ml. of tartaric acid solution (10 per cent.) in Nessler cylinder (b). Dilute both solutions to 50 ml. with water and add 0.3 ml. of thioglycollic acid, followed by 5 ml. of diluted ammonia solution (1 : 1) to the contents of each Nessler cylinder. Stir the solutions and after 5 minutes slowly run the standard iron solution from the burette into Nessler cylinder (a) and an equal amount of water into Nessler cylinder (b), with stirring, until the colours match when the glasses are viewed vertically.

The amount of iron contained in 10 ml. of the "test solution" is equal to the amount of iron contained in the volume of standard iron solution added. Hence the iron oxide content of the sample can be evaluated.

*Volumetric method.* Dilute the "stock solution" to about 350 ml., heat just to boiling point, remove from the source of heat and pass a stream of hydrogen sulphide gas through the solution for 15 minutes. Reheat the solution to 60°C., digest at this temperature for 5 minutes and filter with suction through a paper pulp pad to remove the platinum sulphide. Wash the flask and the precipitate thoroughly with the acid sulphide wash liquid. Reheat the filtrate just to boiling point and pass a stream of hydrogen sulphide gas through it for a further 5 minutes. Add 10 ml. of diluted sulphuric acid (1 : 1), boil under a Bunsen valve until free from hydrogen sulphide (test with lead acetate paper) and continue boiling for a further 10 minutes. Cool under the Bunsen valve and titrate with standard potassium permanganate solution (0.02N).

Evaporate the titrated solution to about 150 ml., cool and dilute to exactly 250 ml. with water to obtain the "stock solution" for the determination of titania.

(ii) *Determination of titania.* Determine the titania colorimetrically with hydrogen peroxide.

NOTE: Instead of the procedure described, suitable colorimeters or absorptimeters may be used. These instruments generally give a greater accuracy and range than do visual determinations.

Fill a burette with standard titanium solution (1 ml. = 0.1 mg.  $\text{TiO}_2$ ) and another with water. Use a pair of matched 100 ml. Nessler cylinders. Pipette 50 ml. of the "stock solution" into Nessler cylinder (a), and add 10 ml. of diluted sulphuric acid (1 : 9) and 40 ml. of water to Nessler cylinder (b) if the iron has been determined colorimetrically, or 20 ml. of diluted sulphuric acid (1 : 9) and 30 ml. of water to Nessler cylinder (b) if the iron has been determined volumetrically. Add 10 ml. of hydrogen peroxide 20 volume to the contents of each Nessler cylinder and stir the solutions. Slowly run the standard titanium solution from the burette into Nessler cylinder (b) and an equal amount of water into Nessler cylinder (a), with stirring, until the colours match when the glasses are viewed vertically.

The amount of titania contained in 50 ml. of the "stock solution" used is equal to the amount of titania in the volume of standard titanium solution used. Hence the titania content of the sample can be evaluated.

(iii) *Calculation of the alumina content.* Subtract the sum of the weights of the ferric oxide and titania from the weight of the mixed oxides from the ammonia precipitation to obtain the weight of alumina in the sample. This weight includes any phosphate in the sample. If required, phosphate may be determined on a separate sample by any standard method.

NOTE. Methods for the direct determination of alumina have been developed, and it is hoped to issue an addendum to this British Standard in due course.

(d) *Determination of lime.* Adjust the volume of the solution reserved for the lime determination to about 250 ml. and add 1 g. of ammonium oxalate. Boil the solution, then add, with stirring, diluted ammonia solution (1 : 1) until the solution is alkaline, followed by an excess of 10 ml. of diluted ammonia solution (1 : 1). Cover the beaker with a clock-glass and digest on a water-bath for 2 hours. Allow to cool and stand, preferably overnight. Filter through a No. 40 Whatman paper (or equivalent) and wash the precipitate 4 times with cold ammonium oxalate solution (1 per cent.). Reserve the filtrate and washings for the determination of magnesia. Wash the precipitate back into the precipitation beaker. Moisten the filter-paper with hot diluted nitric acid (1 : 1) and then wash it thoroughly with hot water, collecting the washings in the precipitation beaker. Reserve the filter paper for ignition. Dissolve the precipitate in hot diluted nitric acid (1 : 1) and dilute the solution to about 80 ml. Add about 0.2 g. of solid ammonium oxalate, boil the solution and precipitate the calcium oxalate as before. Allow to cool and stand at least 3 hours. Filter through a No. 40 Whatman paper (or equivalent) and wash the precipitate thoroughly with cold ammonium oxalate solution (1 per cent.). Combine the filtrate and washings with the solution reserved for the magnesia determination and acidify with hydrochloric acid (sp. gr. 1.16).

Carefully ignite the precipitate and papers in a weighted platinum crucible, slowly at first to burn off the carbon and finally at  $1200^\circ\text{C}$ . for 15 minutes. Allow to cool over a good desiccant as the ignited precipitate is very hygroscopic. Weigh rapidly to obtain the weight of calcium oxide.

(e) *Determination of magnesia.* Evaporate the acidified solution reserved for the magnesia determination to about 300 ml., cool and add 10 ml. of freshly prepared ammonium phosphate solution (10 per cent.). Make the cold solution just alkaline with ammonia solution (sp. gr. 0.880) and stir

vigorously. Add a further 20 ml. of ammonia solution (sp. gr. 0.880), stir vigorously to start the precipitation and allow to stand at least overnight at a temperature below 5°C. Filter through a No. 42 Whatman paper (or equivalent) and wash 4 times with cold diluted ammonia solution (1 : 39). Discard the filtrate and washings. Wash the precipitate back into the precipitation beaker. Moisten the filter paper with hot diluted nitric acid (1 : 1), then wash the paper thoroughly with water, collecting the washings in the precipitation beaker. Dissolve the precipitate in hot diluted nitric acid (1 : 1) and dilute to about 80 ml. with water. Add 1 ml. of freshly prepared ammonium phosphate solution (10 per cent), cool the solution and make it just alkaline with ammonia solution (sp. gr. 0.880). Cool and stir vigorously, then add a further 5 ml. of ammonia solution (sp. gr. 0.880). Stir vigorously again, then allow to stand overnight at a temperature below 5°C. Filter through a No. 42 Whatman paper (or equivalent) or a weighed sintered silica crucible (Grade 4). Wash thoroughly with cold diluted ammonia solution (1 : 39), discarding the filtrate and washings. Ignite the precipitate and paper in a platinum crucible (*see* Note 1), first at a low temperature to burn off the carbon and finally at 1000°C. for 10 minutes, or ignite the sintered silica crucible and precipitate at 1000°C. for 10 minutes. Weigh the residue as magnesium pyrophosphate (*see* Note 2).

$$\text{g. Mg}_2\text{P}_2\text{O}_7 \times 0.3623 = \text{g. MgO.}$$

NOTE 1: Platinum crucibles may be seriously attacked during the ignition if the precipitate is not thoroughly washed with diluted ammonia solution, or if the precipitate is ignited too strongly before all the carbon is oxidised.

NOTE 2: If manganese is detected in the sample for analysis, the weight of magnesium pyrophosphate must be corrected for the manganese pyrophosphate weighed with it. Dissolve the magnesium and manganese pyrophosphates in 20 ml. of diluted sulphuric acid (1 : 1), add 20 ml. of nitric acid (sp. gr. 1.42) and dilute to 90 ml. with water. Add 0.4 g. of potassium periodate, boil for 1 minute, maintain at 90°C. for 10 minutes, cool and dilute to exactly 100 ml. with water. Compare with standards of known manganese content similarly prepared or determine the manganese using a colorimeter or absorptiometer. Calculate the manganese as manganese pyrophosphate and deduct the corresponding weight from the gross weight of the pyrophosphate precipitate.

#### DETERMINATION OF ALKALIS

NOTE: A careful determination of sodium and potassium in the stock of calcium carbonate and ammonium chloride used for the determination of alkalis in samples is essential, and the amounts of sodium and potassium found in the reagents should be subtracted from those found in the analysis of a sample.

16. (a) *Procedure.* Grind 0.5 g. of the finely powdered sample with 0.5 g. of ammonium chloride and then grind the mixture with 3 g. of calcium carbonate in an agate mortar. Place 0.5 g. of calcium carbonate in the bottom of a platinum crucible, preferably of the Lawrence-Smith type, and transfer the mixture from the agate mortar to the crucible. Rinse out the mortar with a further 0.5 g. of calcium carbonate and cover the mixture in the crucible with this portion, consolidating the charge in the crucible by gentle tapping. If an ordinary platinum crucible is used for the sintering, insert it in a hole in an asbestos board so that the lower half projects beneath the board. Cover the crucible with the lid and gently heat until the smell of ammonia is no longer perceptible. If white fumes of ammonium chloride are evolved the temperature is too high. Then heat



for 1 hour in such a way that the lower part of the crucible, that occupied by the lower calcium carbonate layer, is maintained at about  $800^{\circ}\text{C}$ . To render the alkalis soluble and to avoid loss of alkalis and difficulty in extracting the mixture with water, the mass must be sintered but not fused. Allow the crucible to cool and transfer the sintered mass to a platinum or porcelain basin, using hot water to wash the crucible. Crush the sintered mass and digest for 30 minutes with about 80 ml. of hot water. Filter through a No. 40 Whatman paper (or equivalent) and wash the residue twice by decantation with 40 ml. portions of hot water, thoroughly stirring up the residue each time. Transfer the residue to the filter and wash thoroughly with hot water. Discard the residue. (There should be no gritty residue insoluble in hydrochloric acid if the sample has been completely decomposed by the sintering.) Acidify the filtrate with hydrochloric acid, heat to  $90^{\circ}\text{C}$ . and add 1 ml. of barium chloride solution (10 per cent) to remove any sulphate. Just neutralise with diluted ammonia solution (1 : 1) and add 10 ml. of ammonium carbonate solution (20 per cent.) to precipitate the calcium and excess barium. Filter through a No. 40 Whatman paper (or equivalent) and wash the residue with water. Transfer the filtrate to a platinum basin and commence evaporating. Wash the residue back into the precipitation beaker and dissolve in a slight excess of diluted hydrochloric acid (1 : 1), washing the paper thoroughly, first with hydrochloric acid and then with water. Heat the solution to boiling and re-precipitate the calcium and barium carbonates by just neutralising with diluted ammonia solution (1 : 1) and adding 10 ml. of ammonium carbonate solution (20 per cent.). Filter through a No. 40 Whatman paper (or equivalent) and wash the residue thoroughly with a few small portions of water. Discard the residue. Add the filtrate and washings to the solution evaporating in the basin.

Evaporate to dryness, removing the last traces of water by heating in an air oven at  $120^{\circ}\text{C}$ . for 30 minutes. This prevents spitting in the next operation. Gently heat the residue to volatilise the ammonium salts—preferably in an electric furnace at  $450^{\circ}\text{C}$ . If the heating is carried out over a Bunsen burner great care should be taken to avoid local overheating, and consequent loss of alkalis. Treat the contents of the basin with about 3 ml. of ammonium oxalate solution (4 per cent.) and a drop of ammonia solution (sp. gr. 0.880) to dissolve the alkali chlorides and precipitate the remaining calcium. Cover with a clock-glass and allow to stand overnight. Filter through a small No. 50 Whatman paper (or equivalent) into a small platinum basin and wash the residue with cold ammonium oxalate solution (1 per cent.). Discard the residue. Evaporate the filtrate and washings to dryness, partially covering the basin with a watch-glass, if necessary, to avoid loss by spurting. When the residue is completely dry, ignite carefully at  $450^{\circ}\text{C}$ . as before to volatilise ammonium salts. Moisten the cold residue with a few drops of hydrochloric acid (sp. gr. 1.16), evaporate to dryness and carefully ignite at about  $550^{\circ}\text{C}$ ., keeping below the fusion temperature of alkali chlorides. Allow to cool and weigh to obtain the weight of basin plus mixed alkali chlorides. Dissolve the residue in a small amount of hot water and transfer the solution to a small porcelain basin. Reserve for the determination of sodium or potassium. Ignite the platinum basin, cool and weigh. Subtract the weight of the basin from that of the basin plus mixed alkali chlorides. If the residue was not completely water-soluble, filter off the insoluble portion, ignite the

residue and paper and weigh. Subtract the weight of this residue from the weight of the mixed alkali chlorides.

In the reserved solution of mixed alkali chlorides either sodium or potassium may be determined directly, and the weight of the other alkali calculated from the weight of the mixed alkali chlorides.

(b) *Determination of potash and calculation of soda*

NOTE. DANGEROUS EXPLOSIONS may result from the heating of perchloric acid in the presence of carbon, organic material, dust, etc. Evaporations should, therefore, be carried out carefully in a clean fume cupboard.

(i) *Determination of potash.* Add 2 ml. of perchloric acid (sp. gr. 1.20) to the solution of mixed alkali chlorides contained in a small porcelain basin and evaporate on a water-bath until a fuming syrupy mass is obtained. Cool, dilute with 10 ml. of water and evaporate to dryness. To the cold residue add 10 ml. of alcohol mixture A, stir thoroughly and filter through a weighed sintered silica crucible (Grade 4) or asbestos pad. Transfer the residue to the filter using a fine jet of alcohol mixture A and wash 5 times with 5 ml. portions of this reagent. Finally wash twice with 5 ml. portions of alcohol mixture B. Dry for 1 hour at 120°C. and weigh as potassium perchlorate.

$$\text{g. KClO}_4 \times 0.3399 = \text{g. K}_2\text{O}$$

(ii) *Calculation of soda after a potash determination.* The weight of the mixed alkali chlorides and the weight of potassium perchlorate are known. The weight of sodium oxide in the sample can be calculated by means of the following factors:

$$\begin{aligned} \text{g. KClO}_4 \times 0.5381 &= \text{g. KCl} \\ \text{g. NaCl} \times 0.5303 &= \text{g. Na}_2\text{O} \end{aligned}$$

(c) *Determination of soda and calculation of potash*

(i) *Determination of soda.* Transfer the reserved solution of mixed alkali chlorides to a small beaker and evaporate just to dryness. Dissolve in the minimum quantity of water (2 or 3 drops), add 20 ml. of magnesium uranyl acetate solution and stir. Allow to stand for 1 hour at  $20 \pm 1^\circ\text{C}$ . Filter through a sintered glass crucible (Grade 4), and wash the precipitate from the beaker with a fine jet of magnesium uranyl acetate solution. Wash 5 times with 5 ml. portions of alcohol saturated with sodium magnesium uranyl acetate, followed by two 5 ml. portions of ether. Dry the precipitate for 30 minutes at 110°C. and weigh as sodium magnesium uranyl acetate.

$$\text{g. NaMg(UO}_2)_3(\text{C}_2\text{H}_3\text{O}_2)_9 \cdot 6.5\text{H}_2\text{O} \times 0.02058 = \text{g. Na}_2\text{O}$$

(ii) *Calculation of potash after a soda determination.* The weight of the mixed alkali chlorides and the weight of the sodium magnesium uranyl acetate are known. The weight of potassium oxide in the sample can be calculated by means of the following factors:

$$\begin{aligned} \text{g. NaMg(UO}_2)_3(\text{C}_2\text{H}_3\text{O}_2)_9 \cdot 6.5\text{H}_2\text{O} \times 0.0388 &= \text{g. NaCl} \\ \text{g. KCl} \times 0.6317 &= \text{g. K}_2\text{O} \end{aligned}$$

## PART III. STANDARD METHODS OF PHYSICAL TESTING

## STANDARD TEST NO. 2

## THE DETERMINATION OF POROSITY AND DENSITY

## DEFINITIONS

17. For the purposes of this test the following definitions shall apply:

NOTE: The recommended term precedes the definition; other terms that have been used in the same sense are also given but their continued use will lead to confusion.

(a) *Volume*. Unit (metric): millilitre (ml.).

*Bulk volume* ( $V_a$ ). The volume of the solid material plus the volume of the sealed and open pores.

*Apparent solid volume* ( $V_{as}$ ). The volume of the solid material plus the volume of the sealed pores.

*True volume* ( $V_t$ ). The volume of the solid material only. The following terms have also been used: "solid volume" and "volume".

(b) *Density*. Unit (metric): grams per millilitre (g/ml.).

*Bulk Density* ( $S_a$ ). The ratio of the mass of the material to its bulk volume. The term "apparent specific gravity" has also been used to denote this quantity.

*Apparent solid density* ( $S_{as}$ ). The ratio of the mass of a material to its apparent solid volume. The term "apparent true specific gravity" has, on occasion, been used.

*True density* ( $S_t$ ). The ratio of the mass of a material to its true volume. The following terms have also been used: "powder density", "solid density", "absolute density" and "density".

(c) *Specific gravity*. Dimensionless (numerically equal to density when C.G.S. units are employed).

*Bulk specific gravity*. The ratio of the mass of the material to the mass of a quantity of water which at 4°C. has a volume equal to the bulk volume of the material at the temperature of measurement.

*Apparent specific gravity*. The ratio of the mass of the material to the mass of a quantity of water which at 4°C. has a volume equal to the apparent solid volume at the temperature of measurement. The term "apparent true specific gravity" has, on occasion, been used.

*True specific gravity*. The ratio of the mass of the material to the mass of a quantity of water which at 4°C. has a volume equal to the solid volume at the temperature measurement. The following terms have also been used: "powder specific gravity", "absolute specific gravity" and "specific gravity".

(d) *Porosity*. Dimensionless.

*Apparent porosity* ( $P_a$ ). The ratio of the volume of the open pores to the bulk volume of the material.

*True porosity* ( $P_t$ ). The ratio of the volume of the open and sealed pores to the bulk volume of the material.

*Sealed porosity* ( $P_c$ ). The ratio of the volume of the sealed pores to the bulk volume of the material. The term "closed porosity" has also been used.

By definitions:  $P_c = P_t - P_a$ . It is convenient to express these ratios as percentages by use of the following relationships:

$$P_a = 100 \left( 1 - \frac{S_a}{S_{as}} \right) \text{ per cent.}$$

$$P_t = 100 \left( 1 - \frac{S_a}{S_t} \right) \text{ per cent.}$$

$$P_o = 100 S_a \left( \frac{1}{S_{as}} - \frac{1}{S_t} \right) \text{ per cent.}$$

#### A. THE EVACUATION METHOD

(Applicable to all types of refractory)

##### 18. (a) *Apparatus* shall comprise:

Drying oven.

Balance: to weigh to 0.01 g.

Vacuum desiccator.

Evacuating equipment: capable of reducing the pressure to a value not greater than 1 in. of mercury.

Immersion liquid: water may be used unless the test material is unstable in contact with it: paraffin can be used with all types of refractory material but it shall be fractionated before use and the fraction boiling below 200°C. shall be rejected.

(b) *Test material*. At least 3 test pieces shall be cut, broken or ground from the brick to be tested, and their positions in the brick shall be indicated in the report. Each shall have a volume of about 50 to 60 ml. Sharp or friable edges shall be removed by grinding and any loosely adhering grog or dust shall be removed. The test pieces shall be thoroughly dried at a temperature not greater than 110°C. and cooled in a desiccator.

(c) *Test procedure*. The test pieces shall be weighed ( $W_a$ ) and shall then be placed in an empty vacuum desiccator. This weighing and all subsequent weighings shall be made to an accuracy of 0.01 g. The pressure in the desiccator shall be reduced to a value not greater than 1 in. of mercury, and the immersion liquid shall then be slowly admitted, the reduced pressure being maintained until the pieces are covered. The immersed test pieces shall be allowed to stand under reduced pressure for several hours, preferably overnight. Air shall then be allowed to enter the vessel and the test pieces, held in a sling of fine thread, shall be weighed ( $W_b$ ) suspended in the immersion liquid of the same density as that in the desiccator.

Each test piece in turn shall then be lifted slowly from the immersion liquid by means of the sling. The sides of the test piece will drain during this operation and the globules of liquid that form on the underside and between the sling and the top surface shall be removed by brief contact with the edge of a piece of filter paper. Care must be taken that the filter paper does not come into contact with the surface of the test piece since the removal of too much liquid will lead to larger errors than will the removal of insufficient liquid. The soaked test piece shall then be weighed suspended in air ( $W_c$ ).

NOTE: For Method of Calculation see Clause 20.

#### (B) THE HOT TEST PIECE, BOILING WATER METHOD

(HTBW METHOD)

(Applicable to materials of all degrees of porosity if unaffected by immersion in boiling water)

(19) (a) *Apparatus.* The apparatus shall comprise an oven, a tank, preferably thermostatically controlled, a balance, weights and a balance bridge. The oven shall be capable of maintaining a temperature of  $110^{\circ}\text{C}$ . in the volume occupied by the samples being tested. The tank should be fitted with means for introducing cold water below the samples and for removing hot water from above the samples. Any convenient means for the heating of the tank and its contents may be used, such as an immersion heater mounted horizontally near the bottom.

The balance and weights shall be suitable for weighing samples up to 200 g. within  $\pm 0.1$  g. for control tests or within  $\pm 0.01$  g. for referee tests.

(b) *Test material.* At least 3 test pieces shall be cut, broken or ground from the brick to be tested, and their positions in the brick shall be indicated in the report. Each shall have a volume of about 50 to 60 ml. Sharp or friable edges shall be removed by grinding and any loosely adhering grog or dust shall be removed. The test pieces shall be thoroughly dried at a temperature not greater than  $110^{\circ}\text{C}$ . and cooled in a desiccator.

(c) *Test procedure.* The test pieces shall be suspended by loops of cotton of suitable length from rods of suitable material (e.g., stainless steel). The rods and test pieces shall be placed in the oven so that the test pieces hang freely. The oven temperature shall be maintained at  $110^{\circ}\text{C}$ . for several hours, e.g., overnight.

The rods and test pieces shall next be transferred rapidly to the tank containing water at its boiling point. The transfer of each rod with its test pieces should not take longer than about 3 seconds. This is facilitated if the tank is placed adjacent to the oven.

When all the test pieces have been immersed, the boiling shall be continued for 10 minutes. (For some highly porous bricks, metal sinkers should be attached to the test pieces to ensure their complete immersion.)

Heating shall then be discontinued and the hot water displaced by passage of cold water into the bottom of the tank, the test pieces remaining immersed until they have cooled to the prevailing cold water temperature (about 30 minutes in flowing water will suffice).

Each test piece shall then be transferred in turn as quickly as possible from the tank to a suitable container of cold water. (A glass beaker or similar container of about 600 ml. capacity with about 400 ml. of water is suitable.)

The bridge having been put into position over the left-hand balance pan, the container shall be placed on it and the test piece suspended from the balance hook by its loop of cotton thread. (The test piece shall not touch the sides of the container or the surface of the water when the balance beam is raised.)

The weight ( $W_b$ ) of the immersed test piece shall be determined to the nearest 0.1 g. for control and to the nearest 0.01 g. for referee tests.

The container and test piece shall be removed from the balance bridge, the test piece slowly withdrawn from the water by its cotton loop and the residual drops of water below the test piece and between the cotton and the top of the test piece removed by brief contact with the edge of a piece of filter paper. Care must be taken that the filter paper does not touch the surface of the test piece since the removal of too much liquid will lead to larger errors than will the removal of insufficient liquid.

The balance bridge may then be removed and the test piece shall be again suspended from the balance hook and reweighed with the same precision as before. The weight shall be recorded ( $W_e$ ).

After all the test pieces have been weighed in the manner described, they shall be returned to the oven at  $110^{\circ}\text{C}$ . for some hours (conveniently overnight) and re-weighed when cold. The weight shall be recorded ( $W_a$ ). Alternatively,  $W_a$  may be determined before the test pieces have been soaked, but this is less satisfactory with materials that are friable or that have dust in surface pores.

#### CALCULATION OF RESULTS

(20) Whichever method of soaking the test pieces has been employed, the various volumes, densities and porosities shall be calculated from the following formulae:

If  $W_a$  = weight of the dry test piece,

$W_b$  = weight of the test piece soaked with and suspended in the immersion liquid,

$W_c$  = weight of the test piece soaked with the immersion liquid and suspended in air,

$D$  = density of the immersion liquid at the temperature that prevailed during the test,

$S_t$  = true density of the material of the test piece determined by Standard Test No. 3,

then:

$$\text{Bulk volume, } V_a = \frac{W_c - W_b}{D}$$

$$\text{Apparent solid volume, } V_{as} = \frac{W_a - W_b}{D}$$

$$\text{Bulk density, } S_a = \frac{W_a}{W_c - W_b} \times D$$

$$\text{Apparent solid density, } S_{as} = \frac{W_a}{W_a - W_b} \times D$$

$$\text{Apparent porosity, per cent., } P_a = \frac{W_c - W_a}{W_c - W_b} \times 100 = 100 \left( 1 - \frac{S_a}{S_{as}} \right)$$

$$\text{True porosity, per cent., } P_t = 100 \left( 1 - \frac{S_a}{S_t} \right)$$

When C.G.S. units are employed, when water is used as the liquid and weighings are made at a temperature below  $15^{\circ}\text{C}$ .,  $D$  may for all practical purposes be taken as numerically equal to 1. For other temperatures, the following values for the density of water shall be used:

Temperature $^{\circ}\text{C}$ .	15	20	25	30
Density, g/ml.	0.999	0.998	0.997	0.996

## STANDARD TEST No. 3

## THE DETERMINATION OF TRUE SPECIFIC GRAVITY

## (A) THE SPECIFIC GRAVITY BOTTLE METHOD

(To be used for referee purposes. Applicable chiefly to silica refractories)

21. (a) *Apparatus.* Specific gravity bottle complying with B.S. 733:\* a 50 ml. or 25 ml. bottle shall be used. The important feature in the design is that the stopper shall fit, in all positions, in such manner as to leave between the stopper and the neck of the bottle no annular trough in which liquid can collect.

Balance: capable of weighing with an accuracy of at least 0.001 g.

Displacement liquid: a suitable liquid of known specific gravity which must be reasonably stable under a vacuum, *e.g.*, freshly boiled distilled water with the addition of a suitable wetting agent, paraffin distilling above 200°C., decalin, etc.

Vacuum desiccator.

Evacuation equipment: capable of reducing the pressure to a value not greater than 1 in. of mercury.

Thermostatically controlled bath: capable of being maintained at a temperature in the range 25°–30°C. to within  $\pm 0.1^\circ\text{C}$ .

(b) *Test material.* A representative sample of the refractory material shall be ground to pass a 120 mesh B.S. test sieve, and the powder shall be subsequently treated with a magnet if it has been crushed in an iron mortar. The sample shall be dried at 110°C. for 2 hours.

(c) *Test procedure.* In the following description of the procedure, the quantities given assume the use of a 50 ml. bottle; if a 25 ml. bottle is used the quantities shall be halved:

The bottle and stopper shall first be washed and dried and shall then be weighed (a). This and all other weighings shall be made to an accuracy of at least 0.001 g. About 15 g. of the dried test material shall be introduced into the bottle, which shall then be re-weighed (b). 20 to 30 ml. of the displacement liquid shall be introduced into the bottle by means of a pipette.

The bottle and contents shall then be placed in the vacuum desiccator and shall be allowed to stand under reduced pressure (not greater than 1 in. of mercury) for 4 hours. Where conditions permit, the bottle and powder should be placed in the vacuum desiccator and the latter evacuated before the liquid is slowly introduced; the time needed to remove the entrapped air will then be much less than when the bottle is filled under atmospheric pressure. Another expedient that may be used to remove entrapped air is gentle tapping of the desiccator.

After evacuation, the bottle shall be completely filled with the displacement liquid and the stopper carefully inserted so that the excess liquid shall leave by way of the capillary. The temperature of the bottle and its contents shall be below the temperature of the bath during this operation. The bottle shall then be placed in a bath at  $25 \pm 0.1^\circ\text{C}$ . and shall be left there for 30 minutes; if the room temperature should exceed 20°C., the bath temperature shall be raised to at least 5 Centigrade degrees above the room temperature. Any liquid present on the top of the stopper shall be removed before the bottle is taken from the bath; the exterior of the bottle shall then be wiped

\* B.S. 733, "Density bottles."

dry. The bottle, with its contents, shall be transferred to the balance case, allowed to stand there for 30 minutes and shall then be weighed (c).

The bottle shall next be emptied, cleaned and dried: it shall then be completely filled with the displacement liquid. The stopper shall then be inserted. The bottle, suitably weighed, shall be placed in the bath at the same temperature as in the previous operation and left there for 30 minutes. The bottle shall then be carefully dried as before, and, after having been left in the balance case for 30 minutes, shall be weighed (d).

(d) *Calculation of result*

If the weight of the bottle	= a
the weight of the bottle and powder	= b
the weight of the bottle and powder and liquid	= c
the weight of the bottle completely filled with liquid	= d
and the specific gravity of the liquid at the temperature of the bath (compared with that of water at 4°C.)	= G
then the true specific gravity of the powder (compared with that of water at 4°C.)	$= \frac{(b-a)}{(d-a)-(c-b)} \times G.$

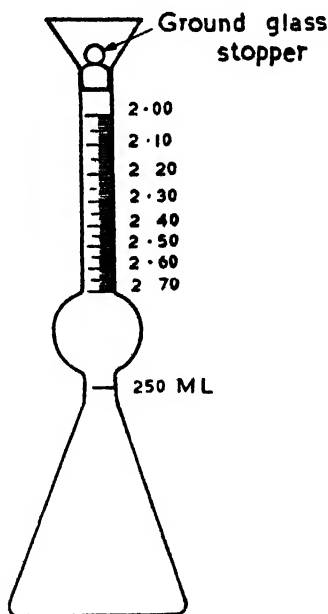


Fig. 1. Rees-Hugill flask

(B) THE REES-HUGILL FLASK METHOD

(Rapid method for control testing, especially of silica refractories)

22. (a) *Apparatus* (see fig. 1). Displacement flask\* of the type designed by W. Hugill and W. J. Rees (*Trans. Brit. Ceram. Soc.*, 24, 70, 1925). Calibration of the flask is essential.

\* A B.S. specification for this type of flask is in course of preparation.



Balance: capable of weighing with an accuracy of at least 0.05 g.

Displacement liquid: xylene.

(b) *Test material.* A representative sample of the dried brick shall be crushed to pass a 30 mesh B.S. test sieve. If the crushing has been carried out in an iron mortar, the powder shall be treated with a magnet. The powder must be at room temperature before it is used in the test.

(c) *Test procedure.* The flask shall be filled, to the lowest mark on the neck, with xylene and shall be allowed to stand for a few minutes to allow any liquid adhering to the neck to drain; the level of the xylene shall be finally adjusted by means of a narrow pipette inserted down the neck of the flask. 100.0 g. of the test material shall be weighed out and introduced into the flask, a little at a time, through a small funnel. When the whole quantity has been introduced, material adhering to the neck of the flask shall be dislodged by gentle tapping. The flask shall then be agitated, without being allowed to become warm by contact with the hands and, after half a minute, the specific gravity of the sample shall be read directly from the graduated neck of the flask.

It is essential that the determination be carried out at a constant temperature, *i.e.*, in a room uniformly heated and free from draughts.

(d) *Order of accuracy.* If a carefully graduated and calibrated flask is used, the order of accuracy of specific gravity determinations by this method is  $\pm 0.005$ .

#### STANDARD TEST NO. 4

##### THE DETERMINATION OF PERMEABILITY TO AIR (Unsuitable for materials of high permeability)

#### DEFINITION

23. The permeability of a material to a gas is defined as the rate at which the gas in question will pass through the material under a definite difference of pressure; permeability, unlike porosity, is a directional property.

#### METHOD OF TEST

24. (a) *Apparatus* (see fig. 2). A simple arrangement is shown in fig. 2. Water from a constant head supply flows into a large bottle† and thence to a large separating funnel\* from which air is driven through the test piece at a pressure indicated by a manometer. The test piece is fixed in a seating in an iron vessel about 10 cm. in diameter; a metal tube leads into the bottom of the vessel and it is essential to ensure that the whole of the lower surface of the test piece is exposed to incoming air. An apparatus of the type used by the British Cast Iron Research Association (*B.C.I.R.A. Spec. Pub. No. 2*, April, 1934) may also be used provided that the time and pressure limits specified in sub-clause (c) below are observed.

(b) *Test material.* The air-dried test piece shall be of square or cylindrical section at least 7 cm. high where the brick allows and at least 25 sq. cm. in cross section. It shall be cut from the brick in such a manner that the height of the test piece is parallel to the direction in which the permeability is to be

† A Winchester bottle is suitable (see B.S.830, "Winchester bottles").

\* A separating funnel of 1000 ml. nominal capacity is suitable.

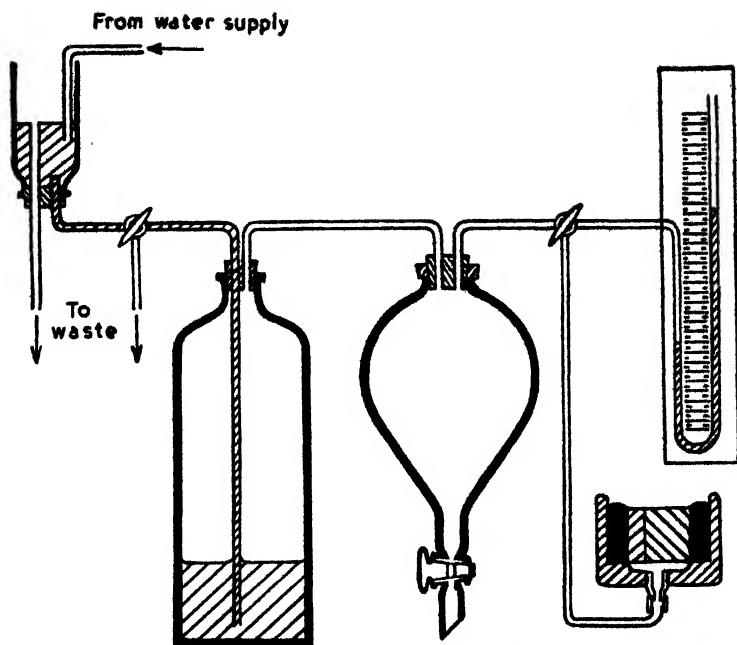


Fig. 2. Permeability apparatus

measured. It shall be stated in the report whether any "skin" is included in the test piece, and whether such a "skin" is perpendicular to the direction of gas flow.

(c) *Test procedure.* The test piece shall be fixed in the iron vessel with sealing wax or modelling clay, and the gap between the walls of the vessel and the test piece shall be filled with mercury. With the type of apparatus described, the further procedure shall be as follows:

A few hundred millilitres of water shall first be siphoned from the large bottle, and water shall then be admitted at a steady rate from a constant head supply. Air will thus be forced through the test piece. The pressure of the air shall not exceed 20 cm. of water. By the time the large bottle is full of water, the rate of flow of air through the specimen should have become constant. The time shall be noted, to the nearest second, when the first drop of water falls into the separating funnel, and manometer readings shall then be taken every half minute. Water shall be allowed to flow into the separating funnel for at least 6 minutes or until at least 100 ml. of water shall have collected in the separating funnel, whichever shall be the longer period. When these conditions have been fulfilled, the time shall again be noted to the nearest second and the flow of water shall simultaneously be stopped. The volume of water in the separating funnel shall be measured to the nearest millilitre; this will be equal to the volume of air that has been driven through the test piece in the measured time. The manometer readings shall be

averaged and the mean pressure under which the air flowed through the test piece will thus be obtained.

(d) *Calculation of results*

If  $V$  = volume of air (ml.) passed through the test piece

$H$  = height of test piece (cm.).

$T$  = time of flow (sec.)

$A$  = area of cross section of test piece (sq. cm.)

$P$  = pressure head under which the air has passed (cm. of water),

the permeability of the sample is equal to  $\frac{V \times H}{T \times A \times P}$  C.G.S. units.

### STANDARD TEST No. 5

#### THE DETERMINATION OF REFRACTORINESS (SQUATTING TEST)

NOTE: The object of this test is to determine the temperature, as indicated by British pyrometric cones, at which a standard pyramidal test piece bends until its tip is level with its base.

#### METHOD OF TEST

25. (a) *Apparatus.* A furnace is required in which an atmosphere containing free oxygen can be maintained throughout the test. It shall be possible to raise the temperature of the test piece and the pyrometric cones at the specified rate to at least 1750°C. The furnace shall have a zone of uniform temperature extending a short distance above and below the test piece.

(b) *Test material.* The test piece, cut from the body of the brick to be tested and not including any of the original face, shall be ground on a suitable abrasive wheel to the shape of a pyramid with a triangular base. One edge of the pyramid shall be perpendicular to the base and  $1\frac{1}{2}$  in. long; the sides of the triangular base shall be  $\frac{1}{2}$  in. A tolerance of  $\frac{1}{16}$  in. shall be allowed in the dimensions of the base; when the base is larger, within the tolerance, the height shall be proportionately larger, and *vice versa*.

It may be found impossible, by the specified method, to prepare a test piece of the stipulated dimensions from certain loosely bonded, highly grogged bricks. Should it be found that the only method of preparing the required test piece is to crush the brick to a powder and mould it with an organic binder, such departure from the standard procedure shall be noted in the report as follows:

"Owing to the friability of the material, the test piece was prepared by crushing the sample and moulding the powder with an organic binder."

(c) *Test procedure.* The test piece shall be mounted at the centre of a refractory disk with a cement consisting of calcined alumina, or well-fired and finely ground sillimanite, bonded with 10 per cent. of china clay. With basic materials, both the disk and the cement material (except that in contact with the pyrometric cones) shall be of a suitable neutral or basic composition. The test piece shall be mounted so that the edge perpendicular to its base shall be vertical. British pyrometric cones, 1 in. high, shall be cemented around the periphery of the disk, oriented so that they shall bend away from the test piece, *i.e.*, with their numbers facing inwards. The edge opposite the numbers shall be vertical.

The test piece with the surrounding pyrometric cones shall be placed in the furnace, and the temperature shall be raised at a steady rate during a period of  $1\frac{1}{2}$  to 2 hours to a temperature estimated to be approximately 200 Centigrade degrees below the squatting temperature; from then onwards the rate of rise of temperature shall be maintained constant at 5 Centigrade degrees per minute.

The test shall be continued till the tip of the test cone shall have bent over level with the base; the disk bearing the specimen shall then be removed from the furnace and the test piece examined when cold.

(d) *Report of the result.* The refractoriness shall be reported as the number of the pyrometric cone that shall have bent over to a similar extent to the test cone. Should it be found that the tip of one pyrometric cone is below the base, whereas the tip of that next in order is above the base, the refractoriness shall be reported as lying between the 2 cone numbers. In the event of the test piece not bending in the normal manner an indication of the type of squatting and of the cone at which it occurs shall be given.

#### STANDARD TEST No. 6

##### THE DETERMINATION OF REFRACTORINESS-UNDER-LOAD

###### GENERAL

26. This method of test describes the procedure for determining the high temperature deformation of refractory materials when subjected to a specified compressive load. Two forms of the test are described:

- (a) *Rising temperature test.* This involves heating the loaded specimen at a prescribed rate until either collapse or a specified amount of deformation occurs.
- (b) *Maintained temperature test.* In this test, the loaded specimen is heated at a prescribed rate to a predetermined temperature, which is maintained for a specified time, or until collapse or a predetermined deformation occurs.

Sections (a) to (f) inclusive apply to both forms of the test.

(a) *Apparatus.* The apparatus shall comprise a suitable furnace and loading device. The furnace may be gas or oil-fired, but the carbon-granule resistance furnace is recommended and is more easily controlled. Where gas or oil-fired furnaces are used, the burners shall be individually controlled and shall fire tangentially to the specimen. Means to prevent flame impingement on the specimen and thermocouple shall be adopted. The furnace shall be capable of being heated at a controlled rate with an oxidising atmosphere, and shall have a zone of uniform temperature extending a short distance above and below the test piece. The furnace shall be fitted with suitable means for applying a pre-determined load to the specimen and for recording the deformation that occurs during the test.

(b) *Temperature measurement.* The temperature up to at least 1250°C. may be measured with a platinum *v.* platinum-rhodium thermocouple suitably protected and placed not more than 1 in. from the centre of one side of the test piece (in carbon-granule furnaces having suitable atmospheres, it is recommended that the thermocouple should have alumina insulators only, and a bare junction that is held in direct contact with the face of the test piece). The temperature indicated by the thermocouple shall be recorded

by means of a galvanometer or recorder of the potentiometer type. From 800°C. upwards until the end of the test, an optical pyrometer shall be sighted on the test piece in such a manner as to ensure adequate observation of the temperature which shall be noted at 5 minute intervals. Temperature readings with the optical pyrometer should be taken on more than one side of the test piece to ensure that it is being evenly heated. British pyrometric cones may also be included on each side of the test piece to give an additional indication of the uniformity of the temperature in the furnace.

(c) *Atmosphere.* The furnace atmosphere shall contain free oxygen.

(d) *Test piece.* The test piece shall be cut or ground to the shape of a prism  $2\frac{1}{2}$  in. high and having a base  $1\frac{3}{4}$  in. square or, alternatively, bored or ground to the shape of a cylinder  $2\frac{1}{2}$  in. high and 2 in. in diameter. The position of the test piece in the original block and the direction in which pressure is applied shall be stated.

Care shall be taken to ensure that the square or circular faces of the test piece are plane and perpendicular to the major axis (height) of the test piece.

The height of the test piece shall be measured before the test along the centre line of each face or at 4 marked points, 90° apart, on a cylindrical test piece. These measurements are preferably made with a micrometer (or vernier callipers) to an accuracy of  $\pm 0.005$  in. (0.125 mm.). All 4 measurements shall be recorded with the mean, which shall be taken as the initial height of the test piece.

(e) *Preparation for the test.* Before the test piece is inserted, the assembled supporting and loading columns shall have been heated under the proposed load to at least the highest temperature to be employed to ensure that no subsidence of the columns occurs. (Since a considerable temperature gradient will normally exist along the columns, it is important that the separate pieces comprising the columns should subsequently be assembled in the same order as that adopted for this preliminary test. This preheating will not, of course, be necessary before each test.)

The test piece, with the  $2\frac{1}{2}$  in. dimension vertical, shall then be placed centrally on the top block of the supporting column (recrystallised silicon carbide or other suitable material). The pieces comprising the loading column (weighed prior to the first test) shall be assembled in sequence centrally on the test piece so that, before the application of the load or any constraining influence, the whole assembly stands central without any tendency to lean or rock. Serious errors may result if the test piece is not loaded uniformly over the whole of its cross-section.

(f) *Test procedure.* (i) *Loading.* A constant load shall be applied to the test piece. For silica bricks this is normally 50 lb. per sq. in. (3.5 kg. per sq. cm.), and for other materials 28 lb. per sq. in. (2 kg. per sq. cm.). Allowance shall be made for the weights of the loading column and other appropriate portions of the loading system in the determination of the adjustment required.

(ii) *Heating.* The test piece shall be heated so that its temperature is raised at a uniform rate of 10 Centigrade degrees per minute from 300°C. upwards.

NOTE: Difficulty in the maintenance of the prescribed rate will frequently be encountered below 300°C. with gas or oil-fired furnaces. It will be sufficient if spalling of the test piece is avoided in this region and control of the heating rate assumed above this temperature. No difficulty in maintenance of the prescribed rate above 100°C. should be experienced with an electric furnace.

The heating schedule depends upon the type of test being made (rising temperature or maintained temperature).

At the completion of the test the load shall be removed and cooling of the furnace shall be facilitated by natural draught.

- (a) *Rising temperature test.* Heating shall be contained at the rate of 10 Centigrade degrees per minute, a record of the expansion/subsidence of the columns and test piece being made either by means of a chart mounted on a drum revolved at a constant convenient rate or by means of a pointer operated by a suitable lever system to magnify the movement of the test piece and thrust column. The movement of the thrust column shall be noted every 5 minutes to coincide with the temperature readings but the actual temperature of failure shall be observed. The heating shall be discontinued when either: (a) the test piece shall have collapsed or (b) a specified deformation shall have occurred (based on the original height of the test piece and measured from the peak of the recorded curve). It is desirable to limit the maximum subsidence to 10 per cent.
- (b) *Maintained temperature test.* Heating shall be continued at the rate of 10 Centigrade degrees per minute until a temperature 50 Centigrade degrees below the intended test temperature shall have been attained. The rate of heating shall then be gradually reduced to a mean value over the 50°C. interval of not less than 5 Centigrade degrees per minute. This reduction of the rate of heating will reduce the risk of overshooting the test temperature and will assist the stabilisation of the furnace at this temperature.

The test temperature shall then be maintained within  $\pm 5^{\circ}\text{C}$ . for a specified period (or until either (a) the test piece shall have collapsed or (b) a specified subsidence, based on the original height of the test piece and measured from the peak of the recorded curve, shall have occurred).

When the subsidence commences before the test temperature is attained, the temperature of the peak of the deformation curve together with the amount of subsidence prior to the attainment of the test temperature shall be reported in addition to that occurring at the test temperature.

(g) *Report of results*

- (a) *Rising temperature test.* The result of the test shall be reported in one of the following forms, accompanied by the recorded temperature/expansion-subsidence curve:
  - (i) under a load of .....lb./sq. in., the sample failed by shear at .....°C.
  - (ii) under a load of .....lb./sq. in., .....per cent. subsidence had occurred at .....°C.
- (b) *Maintained temperature test.* The result of the test shall be reported in one of the following forms, accompanied by the recorded temperature/expansion-subsidence curve:
  - (i) under a load of .....lb./sq. in., the sample failed by shear after .....hours at .....°C.
  - (ii) under a load of .....lb./sq. in., .....per cent. subsidence had occurred after .....hours at .....°C.
  - (iii) under a load of .....lb./sq. in., .....per cent. expansion had occurred after .....hours at .....°C.

## STANDARD TEST NO. 7

THE DETERMINATION OF THE PERMANENT LINEAR CHANGE ON REHEATING  
(Determination of "after-contraction" and "after-expansion")

## METHOD OF TEST

27. (a) *Apparatus.* A furnace is required which can be controlled within the limits set down under the section on "Test procedure". It is essential that an atmosphere is maintained which is continuously oxidising.

Vernier callipers reading to within 0.002 in. (0.05 mm.) or a micrometer gauge may be used for measuring the change in length; for the measurement of the change in volume a volumenometer, of the mercury type for example, is used.

The Vickers-Sugden method of measurement may alternatively be used. In this method (*J. Soc. Glass Tech.*, 1933, 17, 320) the test piece is placed on 3 steel balls on a steel plane; a steel ball on the top of the test piece bears against the plunger of a micrometer dial gauge, which is standardised against steel rods of known lengths.

(b) *Test material.* Specimens shall be cut in more than one direction. If a 9 in.  $\times$  4½ in.  $\times$  3 in. brick is being tested, 3 test pieces shall be taken with their long axes parallel respectively to the length, width and thickness of the brick; if a larger block is being tested, 2 test pieces shall be taken from near the exterior and 2 from near the interior of the block. Each of the test pieces shall be cut or ground to the shape of a prism, as nearly as possible 2½ in.  $\times$  2 in.  $\times$  2 in. in size; the 2½ in.  $\times$  2 in. faces may be left rough, but the 2 faces perpendicular to the length (*i.e.*, the 2 in.  $\times$  2 in. faces) shall be ground plane and parallel.

(c) *Test procedure.* The distance between the parallel faces of the test pieces shall be measured to within 0.002 in. or 0.05 mm.

Alternatively the volume of each test piece may be measured by any suitable method, *e.g.*, a mercury volumenometer.

The test pieces shall be placed in the furnace, suitably shielded from direct flame impingement. The test pieces may be placed in the furnace either horizontally or vertically; they shall rest on a refractory powder and shall be at least half an inch apart to allow free motion of the hot gases. British pyrometric cones may also be included to give an indication of the time-temperature effect in the furnace.

The temperature of the furnace shall be raised at an approximately uniform rate of 5 to 6 Centigrade degrees per minute from 500 Centigrade degrees below the specified temperature until that temperature shall have been attained. (With silica bricks, the rate of heating should be slow until a temperature of 300°C. has been reached.) Provision shall be made for analysis of the atmosphere in the vicinity of the specimens at any time during the test. The testing temperature shall be held constant within  $\pm 10^\circ\text{C}$ . for the specified period, after which the source of heat and air shall be shut off and the furnace cooled 200 Centigrade degrees within 30 minutes and then at its natural rate overnight, the test pieces being allowed to cool in the furnace.

The cold test pieces shall be removed from the furnace and the changes in length or volume shall be determined.

(d) *Report of result.* The linear change of each test piece shall be determined (a) by direct measurement, in which case the increase or decrease in length shall be reported as a percentage of the original length of (b) by calculation of the percentage change in volume, in which case the percentage linear change shall be taken as one-third of the percentage change in volume.

The report should indicate whether the test pieces were fired vertically or horizontally. If any test piece has become warped or bloated, mention of this shall be made in the report.

Where required, evidence of the temperature control and the furnace atmosphere shall be produced.

#### STANDARD TEST No. 8

##### THE DETERMINATION OF COLD CRUSHING STRENGTH

(Test of mechanical strength at normal temperatures. Not applicable to insulating bricks)

##### METHOD OF TEST

28. (a) *Apparatus.* Any mechanical or hydraulic crushing strength machine may be used, provided that one of the platens works on a seating designed to give axial loading.

(b) *Test material.* At least 6 samples shall be taken. Where possible, whole bricks shall be tested. If the size of the bricks permits, no dimension of a test piece shall be less than 3 in.; should the specimens be cut from a large shape they shall be cubes of 3 in. side. Care shall be taken to ensure that the bearing faces of each test piece are plane and perpendicular to the direction of application of the pressure. The test pieces shall be dry.

(c) *Test procedure.* The test piece may be crushed on the flat, on edge, or on end. Tarred roofing felt, of approximately  $\frac{1}{8}$  in. thickness, shall be placed between the bearing faces of the test piece and the platens of the machine. The load shall be applied at a rate of 500 lb. per sq. in. per min. until the test piece has been crushed.

(d) *Report of result.* The crushing strength of a refractory product varies according to the direction in which the pressure is applied. The size of each test piece used and the direction in which the pressure was applied, shall be reported; if the test piece is cut from a larger brick or block, the size of the original brick or block, and the position of the test piece in it, shall be stated.

The mean of at least 6 determinations shall be reported as the crushing strength, in lb. per sq. in. The number of determinations and the range of the individual results shall also be reported.

#### STANDARD TEST No. 9

##### THE DETERMINATION OF REVERSIBLE THERMAL EXPANSION

(a) Dial gauge method for temperatures up to 1200°C. (applicable to all materials)

##### METHOD OF TEST

NOTE: This method involves the measurement of the expansion of the test piece transmitted through a fused silica rod. It is of general application.



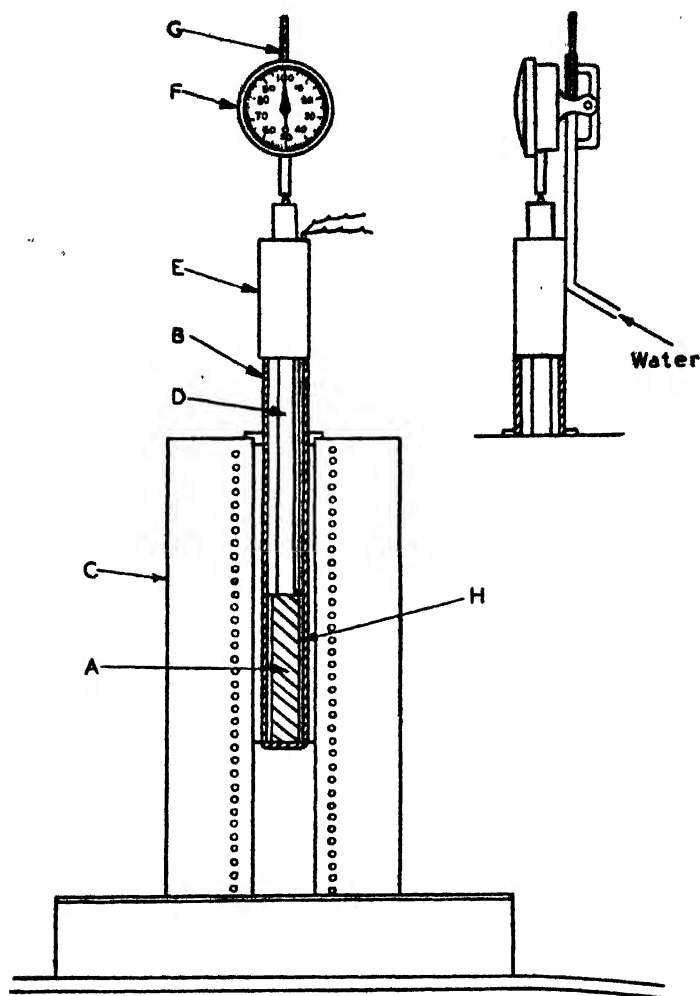


Fig. 3. Dial-gauge apparatus for the determination of reversible thermal expansion  
(Standard test No. 9)

29. (a) *Apparatus.* (i) *Vertical type.* The type of apparatus is illustrated in fig. 3. A suitable furnace (C) consists of a helically grooved refractory tube of  $1\frac{1}{2}$  in. internal diameter, wound with resistance wire and adequately insulated. A fused silica tube (B), about 12 in. long, 1 in. in internal diameter, with an external diameter slightly smaller than the internal diameter of the furnace tube, and with a closed end, rests on a firebrick support inserted in the bottom of the refractory tube; this support is ground to such a length that the test piece (A) is brought up to the high temperature zone of the furnace. Alternatively the fused silica tube (B) can be closed at the lower end

with a refractory disc of equal diameter and about 0.25 in. thick with a deep groove cut across the bottom surface. A piece of nickel-chromium alloy wire 24 in. long is bent twice at right angles so that the centre horizontal part of the wire (1.5 in. long) fits into the groove and the vertical parts run externally along each side of the fused silica tube, the ends protruding well beyond the top of the furnace. While the tube and contents are being lowered into or lifted from the furnace, the ends of the wire are held firmly against the sides of the tube to maintain the disk in position. This arrangement facilitates the removal and insertion of specimens which can be pushed in from the bottom by removing the disk, the tube and its contents first being laid horizontally on a table. A distance rod of fused silica (D), about 10 in. long and 0.5 in. in diameter, rests on the test piece and passes through the metal collar (E), to actuate the micrometer dial gauge (F), which is supported by the collar by means of a water-cooled tube. A thermometer (G) serves to check the constancy of temperature of the metal head. A thermocouple (H) is inserted in the tube so that the hot junction touches the middle of the test piece.

(ii) *Horizontal type.* The apparatus, which can be purchased as a complete unit, is illustrated in fig. 4. It is mounted on a teak base (W), 4 ft.  $\times$  1 ft.  $\times$  2 in. in size. The fused silica tube (AB) is divided by a silica disk (C) fused in at right angles to the axis of the tube. At a point (D) in the tube that is central to the furnace, two holes pierce the top of the tube and through these are threaded the two wires of a platinum-rhodium thermocouple. One end of the tube (B) is fixed into a countersunk recess in the stand (T) and the other (A) is free to move, being supported by an aluminium pulley wheel (P<sub>1</sub>) mounted on ball bearings. Sintered alumina rings a<sub>1</sub> and a<sub>2</sub> are fixed on the tube by means of a mixture of asbestos and china clay, to act as baffles restricting the entrance of cold air into the electric furnace (F).

The test piece (not shown) lies in the tube between disk (C) and the end of the silica rod (XY). This rod just fits in the silica tube (AB) and can move longitudinally without touching the walls, since it is supported from the end (Y), which is fixed into a hole in the Invar steel rod (Z) by means of Wollerton wax. The pulleys P<sub>2</sub> and P<sub>3</sub> (which are mounted on ball bearings in a manner similar to P<sub>1</sub>) carry the Invar rod (Z) which is free to move backwards and forwards with minimum resistance.

The Invar rod transmits the movement of the end of the test piece to a micrometer dial gauge (G), which is bolted in a slot in the iron plate (S).

(iii) *Calibration.* To allow for any temperature variation between the outside silica tube and the specimen, the apparatus shall first be calibrated. With a 10 cm. test piece of fused silica, a calibration test shall be carried out at the standard rate of heating to 1000°C. and any deviation from the zero reading on the dial gauge shall be noted. If the apparatus should be required for use up to 1200°C. the calibration between 1000°C. and 1200°C. shall be obtained by the use of a test piece of sintered alumina instead of fused silica. The linear coefficient of expansion of fused silica is  $0.55 \times 10^{-6}$ , and that of sintered alumina is  $7.9 \times 10^{-6}$ . The expansion figures obtained in all subsequent tests with the apparatus shall be corrected by addition of the difference between the dial reading at a given temperature during the calibration test and the true expansion of 10 cm. of fused silica (or sintered alumina) at that temperature.

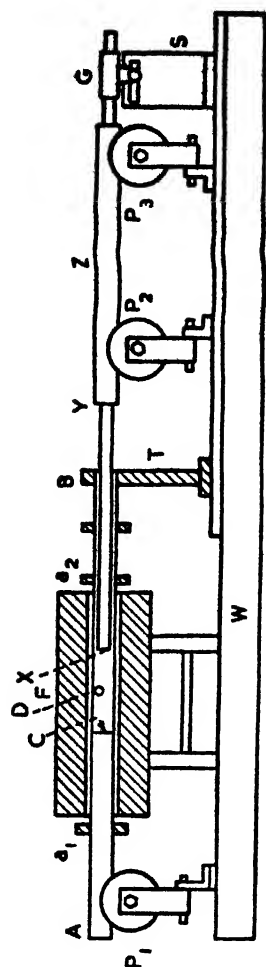


Fig. 4. Horizontal type thermal expansion apparatus

(b) *Test material.* The test piece shall be cut or ground and shall be 10 cm. in length and approximately 2 cm. in diameter. The 2 ends shall be flat and parallel to one another.

(c) *Test procedure.* The length of the test piece shall be accurately measured and it shall then be inserted into the apparatus. The rate of heating of the furnace shall be kept constant at 5 Centigrade degrees per minute. The temperature and dial gauge reading shall be recorded at intervals of 2 minutes (or longer according to the material) until a temperature of 1000°C. or 1200°C. has been attained, when the current shall be switched off and the furnace allowed to cool with the water flowing through the collar of the vertical apparatus. With these types of apparatus it is important to check the

reading on the dial gauge when the specimen has cooled to room temperature. If the needle on the gauge has not returned to within one division on either side of the zero mark the specimen should be re-measured to determine any permanent changes in length which might have occurred during the test. If the zero error cannot be satisfactorily accounted for, the test should be repeated. With unfired and lightly fired specimens some alteration in the size of the specimen must be expected.

(d) *Report of result.* The dial gauge readings, corrected according to the calibration data, shall be calculated to a percentage basis and shall be plotted as a graph relating expansion and temperature. Additional information on the constitution of certain types of refractory materials, notably silica bricks, may be obtained if the coefficient of expansion over small temperature intervals is calculated and plotted.

(b) Telescope method for temperatures up to 1450°C.

#### METHOD OF TEST

NOTE: This method of test describes the procedure for the determination of the reversible thermal expansion of refractory products in the temperature range 750°-1450°C. (Temperatures up to 1550°C. may be attained but the life of the furnace is reduced.)

30. (a) *Apparatus.* The apparatus shall include a horizontal electric tube furnace heated with a platinum coil, a telescope with a vernier scale reading to 0.001 mm. and arranged to sight on each end of the test piece, and a temperature measuring and control unit. It is essential when readings are being taken that the image of each end of the specimen shall always enter the field of the telescope eyepiece from the same side to avoid errors due to any slight whip in the screw thread. Line drawings of a typical apparatus are shown in figs. 5 and 6.

A more convenient apparatus is the comparator consisting of 2 telescopes fixed a predetermined distance apart, the assembly then being capable of traverse in a lateral direction. An additional refinement is a micrometer eyepiece for each telescope.

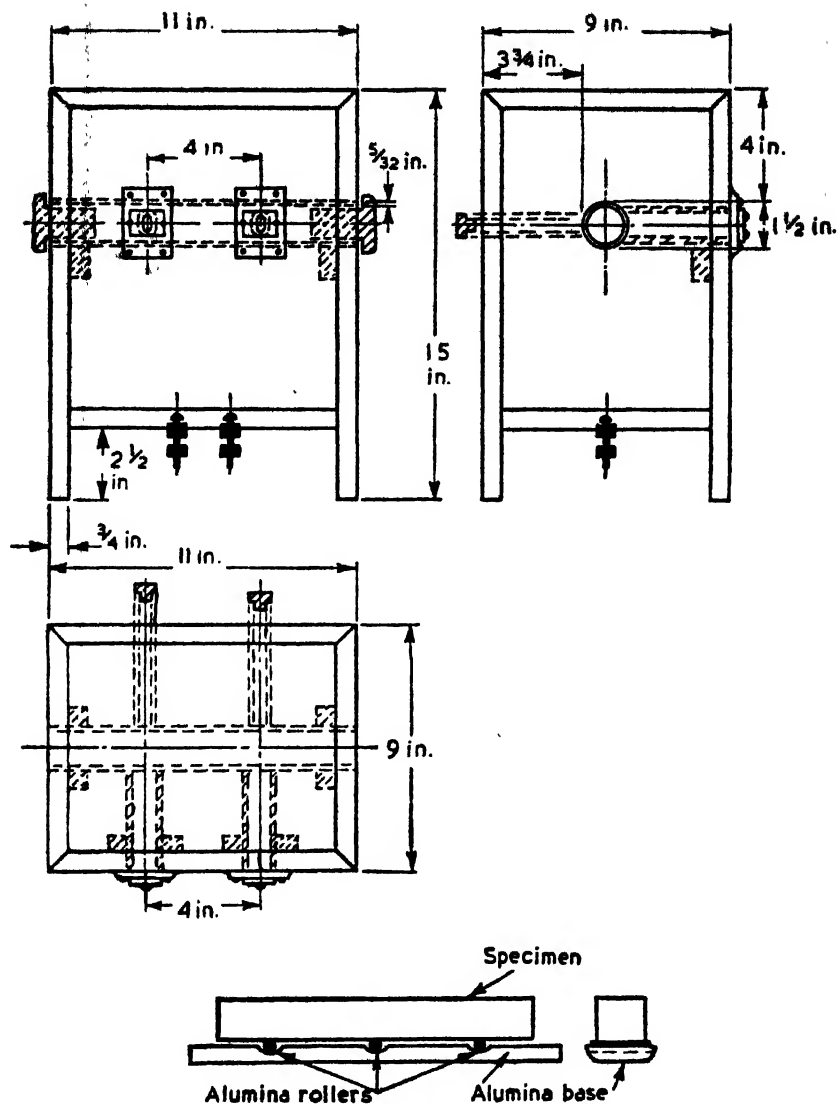
(b) *Temperature measurement.* The temperature shall be measured by a platinum *v.* platinum-rhodium thermocouple suitably protected and placed centrally on the top surface of the test piece. The temperature indicated by the thermocouple shall be recorded, preferably by means of a potentiometer recorder in conjunction with an indicating temperature regulator.

(c) *Atmosphere.* The furnace atmosphere shall contain free oxygen.

(d) *Test material.* The test piece may be cut or ground to the shape of a prism 9.8 cm. in length and 1.5 cm. in cross section. It is preferable to taper the ends away from the edges to be used as sighting edges, thus:

Care shall be taken to ensure that the square ends of the test piece are flat.

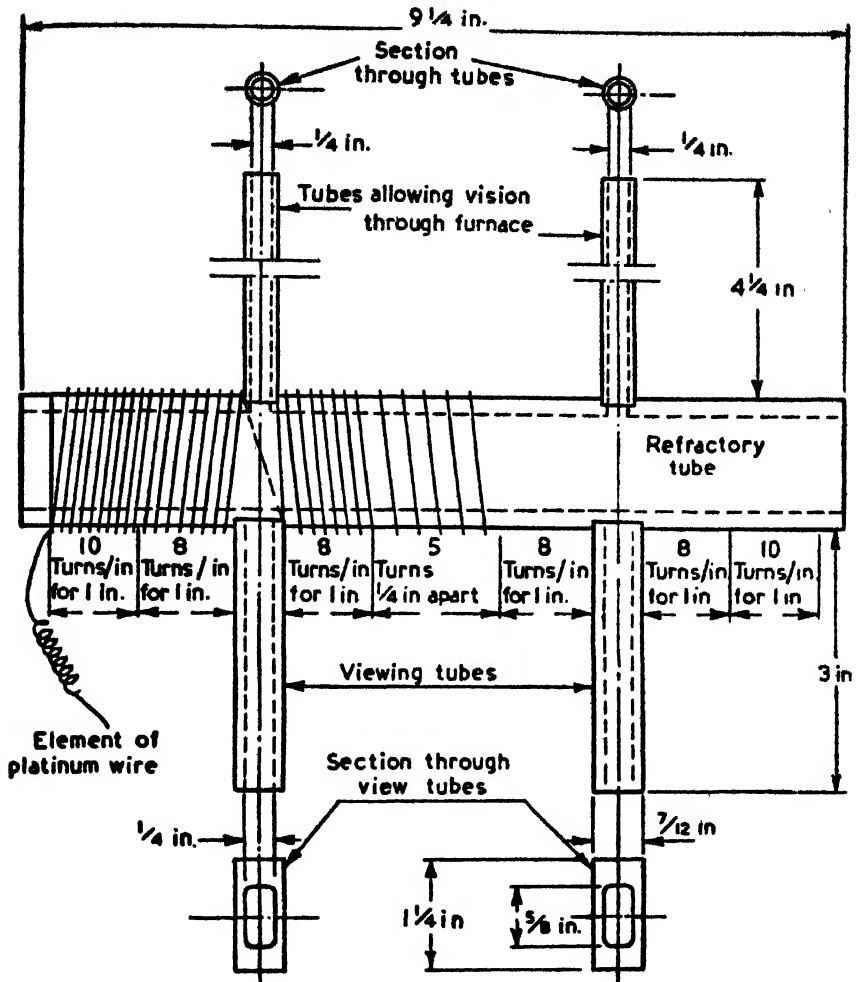
(e) *Preparations for the test.* The test piece shall be mounted on the 3 refractory rollers and shall be inserted centrally into the furnace tube, care being taken to ensure that the test piece moves freely on the rollers and does not touch the side of the tube. The thermocouple shall be inserted and the ends of the furnace tube closed by means of insulating brick stoppers.



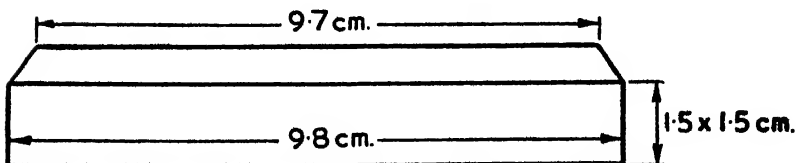
Seating arrangement for specimen

Fig. 5. Thermal expansion furnace sections

(f) *Test procedure.* The length of the test piece shall be measured by means of the vernier telescope, which is sighted on to the ends of the test piece. The readings shall be recorded to the nearest 0.001 mm. The test piece shall be heated so that the temperature as indicated by the thermocouple increases at 10 Centigrade degrees per minute up to 750°C. and from



**Fig. 6. Thermal expansion furnace  
Tube winding and assembly**



**Fig. 7. Test piece**

750° to 1450°C. the heating rate shall be 50 Centigrade degrees per 5 minutes with a 10 minute lag or dwell at the temperature at which expansion readings are being taken.

Readings shall be taken at the desired temperature at the beginning and end of the 10 minutes during which the furnace temperature is constant. (Any wide discrepancy shall be noted and the readings checked.) It is recommended that, when readings are being taken, the travelling telescope shall be moved to a position beyond the end of the test piece and then gradually brought back into position; this will tend to reduce errors due to any slight "whip". It will be found that at temperatures above 1250°C. the test piece becomes more difficult to see; use of a contrasting background such as a piece of white card is advantageous. For normal refractories, readings taken every 50 Centigrade degrees are sufficient, but closer intervals may be used for special work.

(g) *Report of result.* The measured expansions shall be calculated to a percentage basis and shall be plotted as a graph relating expansion and temperature; the report should be accompanied by a time-temperature chart.

#### STANDARD TEST NO. 10

#### THE DETERMINATION OF THE RESISTANCE OF A REFRACTORY MATERIAL TO THE DISINTEGRATING EFFECT OF CARBON MONOXIDE\*

(Applicable to fired refractory products, especially for use in upper parts of blast-furnace linings)

#### METHOD OF TEST

31. (a) *Apparatus.* A wire-wound furnace suitable for use at 450° to 500°C. is required. A source of carbon monoxide, a manometer, a flowmeter and means for the disposal of carbon monoxide are also needed. The furnace tube may be of heat-resisting glass to permit easy observation of the test pieces.

(b) *Test material.* Test pieces shall be cut from the centre and from the exterior of the sample, and shall be ground to the approximate shape of cylinders about 2 in. long and 1.5 in. in diameter.

(c) *Test procedure.* Carbon monoxide, taken from a gas cylinder or generated in the laboratory from formic acid and sulphuric acid, or by the passage of carbon dioxide from a cylinder over charcoal heated to 1000°C. in a tube furnace, shall be passed at a constant rate of 2 l. per hr. through a purifying train to remove carbon dioxide, oxygen and water vapour. Nitrogen used for purging shall be purified in the same way. The water vapour shall be removed by magnesium perchlorate, silica gel or phosphorus pentoxide but not by sulphuric acid or calcium chloride. A manometer and flowmeter shall also be included in the gas train prior to the entrance of the gas into the furnace. The test pieces shall be placed in the furnace tube, and be heated to 450°C. in a stream of nitrogen; carbon monoxide shall then be passed, and the test pieces shall be examined from time to time for general discoloration, carbon deposition at separate nuclei, and cracking. The test shall be continued for 200 hours or until the test pieces shall have disintegrated, whichever is the shorter period; only the time during which the hot pieces are exposed to the stream of carbon monoxide shall be reckoned.

\* Warning: In the use of carbon monoxide, proper precautions for the protection of laboratory personnel should be taken.

(d) *Report of result.* The result shall be reported in the following terms for each test piece:

- (i) The test piece was not disintegrated after.....hours' exposure to carbon monoxide at  $450^{\circ}$  to  $500^{\circ}\text{C}$ .
- (ii) Carbon deposition was noticed after.....hours' exposure to carbon monoxide at  $450^{\circ}$  to  $500^{\circ}\text{C}$ . and the test piece had disintegrated after a total treatment of.....hours.

A short description of the appearance of each test piece after the test shall also be given.

## APPENDIX

### THE DETERMINATION OF RESISTANCE TO THERMAL SHOCK (SPALLING)

(For information only)

In the present state of knowledge of the fundamental factors that control the tendency of material to crack when subjected to thermal shock, it is undesirable to put forward any simulative test as a standard method. The following "small prism" test is considered useful for general purposes:

1. *Apparatus.* A furnace of the muffle or semi-muffle type is needed. It shall be of such a size as to allow sufficient heat release to the test pieces. To accommodate 3 test pieces, the furnace chamber shall be at least 2 ft.  $\times$  1 ft.  $\times$  1 ft. in size; if a large number of specimens are to be tested, the furnace shall be of such a size that the fall in temperature when the test pieces are inserted after the first cooling shall not exceed 20 Centigrade degrees. A thermocouple is required for the measurement of the temperature.

2. *Test material.* Three test pieces shall be cut or ground to the shape of prisms 3 in. high and with a square base of 2 in. side. The test pieces shall be thoroughly dried before being tested.

3. *Test procedure.* The test pieces shall be placed in the cold furnace. The furnace shall then be heated at a uniform rate so that in 3 hours it attains a temperature of  $450^{\circ}\text{C}$ . if silica bricks are being tested, or  $1000^{\circ}\text{C}$ . if firebricks, siliceous bricks or basic bricks are being tested. The testing temperature shall be maintained for 30 minutes and the test pieces shall then be removed from the furnace with a pair of light tongs, which shall have been warmed in the furnace for a short time before use. The test pieces shall be placed on end on a brick floor in a position free from draughts. After they have been cooled in this way for 10 minutes the test pieces shall be replaced in the furnace (which shall have been maintained at the temperature of the test) for a further 10 minutes and the cycle shall then be repeated. Towards the end of each 10 minute cooling period, the test pieces shall be examined. The test shall be concluded when the specimens can be pulled apart in the hands while hot.

4. *Report of the result.* The number of complete cycles of heating and cooling required to promote fracture shall be reported together with a note of the cycle during which cracks first appeared. The 3 individual results shall be included in the report.



# APPENDIX 2I

## FOUR-FIGURE LOGARITHMS

N											Proportional Parts									
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9	
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	*4	8	12	17	21	25	29	33	37	
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34	
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31	
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29	
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27	
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	*3	6	8	11	14	17	20	22	25	
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24	
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22	
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21	
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20	
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19	
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18	
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17	
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17	
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16	
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15	
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15	
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14	
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14	
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13	
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13	
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12	
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12	
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12	
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11	
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11	
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11	
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10	
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10	
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10	
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10	
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9	
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9	
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9	
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9	
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9	
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	8	9	
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	6	7	8	9	
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	5	6	7	8	9	
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	5	6	7	8	9	
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8	
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8	
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	3	3	4	5	6	7	7	
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	3	3	4	5	6	6	7	
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	3	3	4	5	6	6	7	
N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9	

\* Interpolation in this section of the table is inaccurate.

## APPENDIX 21

## FOUR-FIGURE LOGARITHMS—continued

N											Proportional Parts									
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9	
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7	
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7	
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7	
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7	
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7	
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	1	1	2	3	4	4	5	6	6	
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6	
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6	
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6	
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6	
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6	
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6	
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6	
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6	
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6	
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6	
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5	
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5	
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5	
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5	
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5	
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5	
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5	
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5	
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9025	1	1	2	2	3	3	4	4	5	
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5	
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5	
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5	
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5	
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5	
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5	
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5	
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4	
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4	
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4	
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4	
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4	
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4	
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4	
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4	
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	0	1	1	2	2	3	3	4	4	
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4	
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4	
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4	
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	4	4	
N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9	

APPENDIX 2 I  
ANTI- LOGARITHMS

	0 1 2 3 4					5 6 7 8 9					Proportional Parts 1 2 3 4 5 6 7 8 9								
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
.00	1000	1002	1005	1007	1009	1012	1014	1016	1019	1021	0	0	1	1	1	1	2	2	2
.01	1023	1026	1028	1030	1033	1035	1038	1040	1042	1045	0	0	1	1	1	1	2	2	2
.02	1047	1050	1052	1054	1057	1059	1062	1064	1067	1069	0	0	1	1	1	1	2	2	2
.03	1072	1074	1076	1079	1081	1084	1086	1089	1091	1094	0	0	1	1	1	1	2	2	2
.04	1096	1099	1102	1104	1107	1109	1112	1114	1117	1119	0	1	1	1	1	2	2	2	2
.05	1122	1125	1127	1130	1132	1135	1138	1140	1143	1146	0	1	1	1	1	2	2	2	2
.06	1148	1151	1153	1156	1159	1161	1164	1167	1169	1172	0	1	1	1	1	2	2	2	2
.07	1175	1178	1180	1183	1186	1189	1191	1194	1197	1199	0	1	1	1	1	2	2	2	2
.08	1202	1205	1208	1211	1213	1216	1219	1222	1225	1227	0	1	1	1	1	2	2	2	3
.09	1230	1233	1236	1239	1242	1245	1247	1250	1253	1256	0	1	1	1	1	2	2	2	3
.10	1259	1262	1265	1268	1271	1274	1276	1279	1282	1285	0	1	1	1	1	2	2	2	3
.11	1288	1291	1294	1297	1300	1303	1306	1309	1312	1315	0	1	1	1	2	2	2	2	3
.12	1318	1321	1324	1327	1330	1334	1337	1340	1343	1346	0	1	1	1	2	2	2	2	3
.13	1349	1352	1355	1358	1361	1365	1368	1371	1374	1377	0	1	1	1	2	2	2	2	3
.14	1380	1384	1387	1390	1393	1396	1400	1403	1406	1409	0	1	1	1	2	2	2	2	3
.15	1413	1416	1419	1422	1426	1429	1432	1435	1439	1442	0	1	1	1	2	2	2	2	3
.16	1445	1449	1452	1455	1459	1462	1466	1469	1472	1476	0	1	1	1	2	2	2	2	3
.17	1479	1483	1486	1489	1493	1496	1500	1503	1507	1510	0	1	1	1	2	2	2	2	3
.18	1514	1517	1521	1524	1528	1531	1535	1538	1542	1545	0	1	1	1	2	2	2	2	3
.19	1549	1552	1556	1560	1563	1567	1570	1574	1578	1581	0	1	1	1	2	2	2	2	3
.20	1585	1589	1592	1596	1600	1603	1607	1611	1614	1618	0	1	1	1	2	2	2	2	3
.21	1622	1626	1629	1633	1637	1641	1644	1648	1652	1656	0	1	1	2	2	2	2	2	3
.22	1660	1663	1667	1671	1675	1679	1683	1687	1690	1694	0	1	1	2	2	2	2	2	3
.23	1698	1702	1706	1710	1714	1718	1722	1726	1730	1734	0	1	1	2	2	2	2	2	3
.24	1738	1742	1746	1750	1754	1758	1762	1766	1770	1774	0	1	1	2	2	2	2	2	3
.25	1778	1782	1786	1791	1795	1799	1803	1807	1811	1816	0	1	1	2	2	2	2	2	3
.26	1820	1824	1828	1832	1837	1841	1845	1849	1854	1858	0	1	1	2	2	2	2	2	3
.27	1862	1866	1871	1875	1879	1884	1888	1892	1897	1901	0	1	1	2	2	2	2	2	3
.28	1905	1910	1914	1919	1923	1928	1932	1936	1941	1945	0	1	1	2	2	2	2	2	3
.29	1950	1954	1959	1963	1968	1972	1977	1982	1986	1991	0	1	1	2	2	2	2	2	3
.30	1995	2000	2004	2009	2014	2018	2023	2028	2032	2037	0	1	1	2	2	2	2	2	3
.31	2042	2046	2051	2056	2061	2065	2070	2075	2080	2084	0	1	1	2	2	2	2	2	3
.32	2089	2094	2099	2104	2109	2113	2118	2123	2128	2133	0	1	1	2	2	2	2	2	3
.33	2138	2143	2148	2153	2158	2163	2168	2173	2178	2183	0	1	1	2	2	2	2	2	3
.34	2188	2193	2198	2203	2208	2213	2218	2223	2228	2234	1	1	2	2	2	2	2	2	3
.35	2239	2244	2249	2254	2259	2265	2270	2275	2280	2286	1	1	2	2	2	2	2	2	3
.36	2291	2296	2301	2307	2312	2317	2323	2328	2333	2339	1	1	2	2	2	2	2	2	3
.37	2344	2350	2355	2360	2366	2371	2377	2382	2388	2393	1	1	2	2	2	2	2	2	3
.38	2399	2404	2410	2415	2421	2427	2432	2438	2443	2449	1	1	2	2	2	2	2	2	3
.39	2455	2460	2466	2472	2477	2483	2489	2495	2500	2506	1	1	2	2	2	2	2	2	3
.40	2512	2518	2523	2529	2535	2541	2547	2553	2559	2564	1	1	2	2	2	2	2	2	3
.41	2570	2576	2582	2588	2594	2600	2606	2612	2618	2624	1	1	2	2	2	2	2	2	3
.42	2630	2636	2642	2649	2655	2661	2667	2673	2679	2685	1	1	2	2	2	2	2	2	3
.43	2692	2698	2704	2710	2716	2723	2729	2735	2742	2748	1	1	2	2	2	2	2	2	3
.44	2754	2761	2767	2773	2780	2786	2793	2799	2805	2812	1	1	2	2	2	2	2	2	3
.45	2818	2825	2831	2838	2844	2851	2858	2864	2871	2877	1	1	2	2	2	2	2	2	3
.46	2884	2891	2897	2904	2911	2917	2924	2931	2938	2944	1	1	2	2	2	2	2	2	3
.47	2951	2958	2965	2972	2979	2985	2992	2999	3006	3013	1	1	2	2	2	2	2	2	3
.48	3020	3027	3034	3041	3048	3055	3062	3069	3076	3083	1	1	2	2	2	2	2	2	3
.49	3090	3097	3105	3112	3119	3126	3133	3141	3148	3155	1	1	2	2	2	2	2	2	3
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9

## APPENDIX

705

APPENDIX 21  
ANTI-LOGARITHMS—continued

	0 1 2 3 4					5 6 7 8 9					Proportional Parts 1 2 3 4 5 6 7 8 9									
.50	3162	3170	3177	3184	3192	3199	3206	3214	3221	3228	1	1	2	3	4	4	5	6	7	
.51	3236	3243	3251	3258	3266	3273	3281	3289	3296	3304	1	2	2	3	4	5	5	6	7	
.52	3311	3319	3327	3334	3342	3350	3357	3365	3373	3381	1	2	2	3	4	5	5	6	7	
.53	3388	3396	3404	3412	3420	3428	3436	3443	3451	3459	1	2	2	3	4	5	6	6	7	
.54	3467	3475	3483	3491	3499	3508	3516	3524	3532	3540	1	2	2	3	4	5	6	6	7	
.55	3548	3556	3565	3573	3581	3589	3597	3606	3614	3622	1	2	2	3	4	5	6	7	7	
.56	3631	3639	3648	3656	3664	3673	3681	3690	3698	3707	1	2	3	3	4	5	6	7	8	
.57	3715	3724	3733	3741	3750	3758	3767	3776	3784	3793	1	2	3	3	4	5	6	7	8	
.58	3802	3811	3819	3828	3837	3846	3855	3864	3873	3882	1	2	3	4	4	5	6	7	8	
.59	3890	3899	3908	3917	3926	3936	3945	3954	3963	3972	1	2	3	4	5	5	6	7	8	
.60	3981	3990	3999	4009	4018	4027	4036	4046	4055	4064	1	2	3	4	5	6	6	7	8	
.61	4074	4083	4093	4102	4111	4121	4130	4140	4150	4159	1	2	3	4	5	6	7	8	9	
.62	4169	4178	4188	4198	4207	4217	4227	4236	4246	4256	1	2	3	4	5	6	7	8	9	
.63	4266	4276	4285	4295	4305	4315	4325	4335	4345	4355	1	2	3	4	5	6	7	8	9	
.64	4365	4375	4385	4395	4406	4416	4426	4436	4446	4457	1	2	3	4	5	6	7	8	9	
.65	4467	4477	4487	4498	4508	4519	4529	4539	4550	4560	1	2	3	4	5	6	7	8	9	
.66	4571	4581	4592	4603	4613	4624	4634	4645	4656	4667	1	2	3	4	5	6	7	9	10	
.67	4677	4688	4699	4710	4721	4732	4742	4753	4764	4775	1	2	3	4	5	7	8	9	10	
.68	4786	4797	4808	4819	4831	4842	4853	4864	4875	4887	1	2	3	4	6	7	8	9	10	
.69	4898	4909	4920	4932	4943	4955	4966	4977	4989	5000	1	2	3	5	6	7	8	9	10	
.70	5012	5023	5035	5047	5058	5070	5082	5093	5105	5117	1	2	4	5	6	7	8	9	11	
.71	5129	5140	5152	5164	5176	5188	5200	5212	5224	5236	1	2	4	5	6	7	8	10	11	
.72	5248	5260	5272	5284	5297	5309	5321	5333	5346	5358	1	2	4	5	6	7	9	10	11	
.73	5370	5383	5395	5408	5420	5433	5445	5458	5470	5483	1	3	4	5	6	8	9	10	11	
.74	5495	5508	5521	5534	5546	5559	5572	5585	5598	5610	1	3	4	5	6	8	9	10	12	
.75	5623	5636	5649	5662	5675	5689	5702	5715	5728	5741	1	3	4	5	7	8	9	10	12	
.76	5754	5768	5781	5794	5808	5821	5834	5848	5861	5875	1	3	4	5	7	8	9	11	12	
.77	5888	5902	5916	5929	5943	5957	5970	5984	5998	6012	1	3	4	5	7	8	10	11	12	
.78	6026	6039	6053	6067	6081	6095	6109	6124	6138	6152	1	3	4	6	7	8	10	11	13	
.79	6166	6180	6194	6209	6223	6237	6252	6266	6281	6295	1	3	4	6	7	9	10	11	13	
.80	6310	6324	6339	6353	6368	6383	6397	6412	6427	6442	1	3	4	6	7	9	10	12	13	
.81	6457	6471	6486	6501	6516	6531	6546	6561	6577	6592	2	3	5	6	8	9	11	12	14	
.82	6607	6622	6637	6653	6668	6683	6699	6714	6730	6745	2	3	5	6	8	9	11	12	14	
.83	6761	6776	6792	6808	6823	6839	6855	6871	6887	6902	2	3	5	6	8	9	11	13	14	
.84	6918	6934	6950	6966	6982	6998	7015	7031	7047	7063	2	3	5	6	8	10	11	13	15	
.85	7079	7096	7112	7129	7145	7161	7178	7194	7211	7228	2	3	5	7	8	10	12	13	15	
.86	7244	7261	7278	7295	7311	7328	7345	7362	7379	7396	2	3	5	7	8	10	12	13	15	
.87	7413	7430	7447	7464	7482	7499	7516	7534	7551	7568	2	3	5	7	9	10	12	14	16	
.88	7586	7603	7621	7638	7656	7674	7691	7709	7727	7745	2	4	5	7	9	11	12	14	16	
.89	7762	7780	7798	7816	7834	7852	7870	7889	7907	7925	2	4	5	7	9	11	13	14	16	
.90	7943	7962	7980	7998	8017	8035	8054	8072	8091	8110	2	4	6	7	9	11	13	15	17	
.91	8128	8147	8166	8185	8204	8222	8241	8260	8279	8299	2	4	6	8	9	11	13	15	17	
.92	8318	8337	8356	8375	8395	8414	8433	8453	8472	8492	2	4	6	8	10	12	14	15	17	
.93	8511	8531	8551	8570	8590	8610	8630	8650	8670	8690	2	4	6	8	10	12	14	16	18	
.94	8710	8730	8750	8770	8790	8810	8831	8851	8872	8892	2	4	6	8	10	12	14	16	18	
.95	8913	8933	8954	8974	8995	9016	9036	9057	9078	9099	2	4	6	8	10	12	15	17	19	
.96	9120	9141	9162	9183	9204	9226	9247	9268	9290	9311	2	4	6	8	11	13	15	17	19	
.97	9333	9354	9376	9397	9419	9441	9462	9484	9506	9528	2	4	7	9	11	13	15	17	19	
.98	9550	9572	9594	9616	9638	9661	9683	9705	9727	9750	2	4	7	9	11	13	16	18	20	
.99	9772	9795	9817	9840	9863	9886	9908	9931	9954	9977	2	5	7	9	11	14	16	18	20	
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9	



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*Addendum*

REFRACTORIES  
FOR OXYGEN STEELMAKING

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THE FACT THAT A substantial addition is necessary to a book fully revised only five years ago is a measure of the rate of change that has accompanied the arrival of tonnage oxygen. The use of oxygen in steelmaking is not new, but its availability at only a few shillings per thousand cubic feet when made in quantities of 100 tons or more per day, has led to substantial changes both in steelmaking and refractory practice. In particular it has led to new processes, such as the LD, that enable steel of open-hearth quality to be made in a fraction of the normal time and with greatly reduced consumption of refractories. At present these new processes only account for a small part of the world's steel production, *viz.*, about 30 million tons per annum, but this is expected to increase within the next few years to at least 70 million tons. Until recently roughly 90 per cent. of the steel made in Great Britain was produced in open-hearth furnaces, the remaining 10 per cent. being equally divided between pneumatic processes and electric furnaces. Although the early demise of the open hearth, predicted in some quarters, now seems improbable, the balance is likely to shift quite substantially.

Reasons why the open-hearth furnace is likely to survive have been summarised by A. Jackson as follows:

- (1) High flexibility, particularly as regards scrap/pig percentage.
- (2) The great variety of steels it can make.
- (3) High yield.
- (4) The increasing output rates made possible by high oxygen usage.
- (5) Lower costs with larger furnaces.
- (6) Substantial capital investment.

Dramatic support has been given to these views by the Steel Company of Canada, where A. K. Moore reports average outputs of over 100 tons per hour in a campaign on a new 500-ton all-basic furnace. Crystal-gazing is always hazardous but it is probably safe to say that the traditional distribution in this country of roughly 90-5-5 for open-hearth, pneumatic and electric processes, may well change to 60-25-15, or even 50-30-20 within a decade or so.

Speed, whether in a motor-car or a furnace, must be paid for. If more fuel is burned wear rates tend to increase, though output gains may well offset additional costs. In an earlier analysis of all-basic furnaces it was suggested that an increase from 38 to 76 lb. per sq. ft. per hour was associated with a drop in roof life from 1500 to 500 heats. Later experience confirms that faster driving means more wear, but overall costs are nevertheless frequently lower than with silica furnaces, due to reduced overheads and, in some cases, savings in fuel.

In the processes described below, the trend is always towards faster driving, achieved by the use of oxygen, either to intensify combustion or accelerate metalloïd removal from molten iron. To the refractories' technologist such operations immediately suggest three major risks, *viz.*, higher temperatures, increased flux concentration, and alternate reducing and oxidising atmospheres. Refractory life under these arduous conditions is important, not only because it determines refractories costs per ton of steel, but because it controls furnace availability—a vital matter with such large tonnage units. Thus an improvement in the lining life of a converter from, say, 50 to 100 heats, may well mean that two vessels can be used where previously three were required.

#### (a) TEMPERATURE

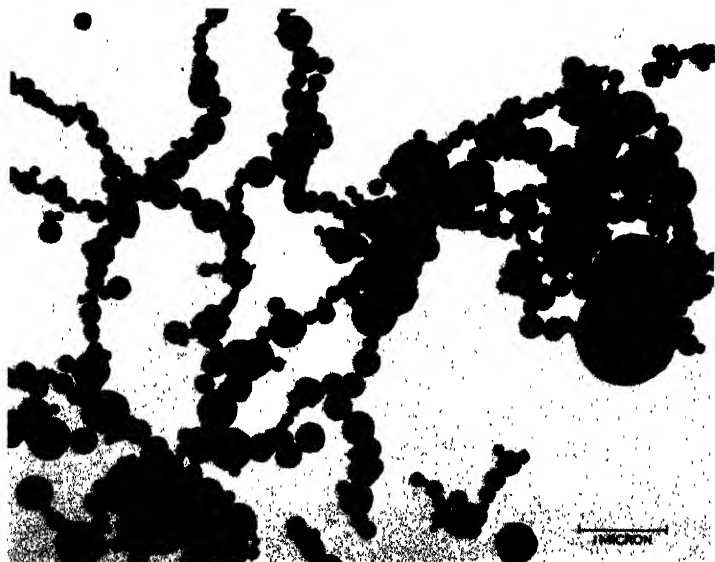
The theoretical flame temperature for oil burned with cold air is approximately 2000°C.: burned with cold oxygen it would be nearer 2500°C. Since oil flames even when fed with cold air can melt silica bricks a potential increase of 500°C. is quite serious. In open-hearth furnaces oil is usually burned with air at nearer 1000°C., corresponding to a theoretical flame temperature of about 2350°C. With oxygen preheated to 1000°C. this figure would rise to nearer 2600°C. The maximum safe operating level with conventional open-hearth furnaces is generally taken as about 1650°C. This is roughly 60°C. below the melting point of pure silica but only 30°C. above the melting point of the working face of a used brick—a modest safety factor bearing in mind the difficulties of roof pyrometry and furnace design. Where oxygen impinges on a bath of molten iron, far higher temperatures—probably in the range 2500–3500°C—may be expected. The salvation of refractories under such conditions lies in keeping them as far away from the hot spot as possible—a principal feature of the new processes described below.

#### (b) FLUXES

Most of the wear on open-hearth furnaces with silica roofs is explainable in terms of solution by iron oxide. Where oxygen lancing is employed, particularly at high carbon levels, evolution of iron oxide is



*Fig. A1. Iron oxide plume shortly after lancing with oxygen in open-hearth furnace.*



*Fig. A2. Electron microscope photograph of iron oxide fume showing chains of magnetic gamma hematite particles.*





*Fig. A3. Modest boil in oxygen lanced open-hearth furnace bath.*

vastly increased (*see* fig. A1, page 713) chemical analysis, together with X-ray and microscopic examination, suggesting that these fumes consist in the main of sub-micron size particles of  $\gamma$  hematite (*see* fig. A2, p. 713.) The mechanism of fume formation is still far from clear, though the evidence now strongly suggests the volatilisation of metallic iron and subsequent oxidation in the furnace atmosphere. The presence of carbon, either in the fuel or in the melt, tends to increase fume level. Turkdogan, Grievson and Darken, have recently suggested that this is because carbon prevents the formation of a relatively impervious oxide skin. Basic refractories show far higher resistance to iron oxide attack than silica, magnesia being among the most resistant materials known. For many purposes, however, dolomite may well prove a competitor, both because it is cheaper and because it is proving more durable under such conditions than would be expected from simple theory or prior experience. The replacement of dolomite by magnesite in open-hearth furnace bottoms making low carbon steels has been shown to cut settling costs to as little as one third. This is in line with calculations made by Ford and White in 1958, who found the solubility of magnesia and doloma in dicalcium ferrite slag to vary with temperature in the manner shown in Table A1.

TABLE A1

PARTS OF REFRACTORY DISSOLVED BY 100 PARTS OF  $2\text{CaO}.\text{Fe}_2\text{O}_3$   
SLAG

<i>Temp. °C.</i>	<i>Magnesia</i>	<i>Doloma</i>	$\frac{\text{Doloma Dissolved}}{\text{Magnesia Dissolved}}$
1700	29	79	2.7
1800	38	105	2.7
1900	50	144	2.9
2000	67	208	3.1

It will be seen not only that magnesia is superior but that its superiority increases with rising temperature. In a paper included in the recent Iron and Steel Institute Special Report No. 74, White suggests a possible explanation of the anomaly in terms of phase diagrams. Attack by slag is not, however, the full story, for in many positions, *e.g.*, open-hearth furnace roofs, spalling resistance, volume stability and hot strength may be equally important. It is probably for this reason

that chrome-magnesite or magnesite-chrome bricks are generally preferred to straight magnesite.

(c) ATMOSPHERE

At certain temperatures alternate oxidising and reducing conditions are known to have a serious effect on basic refractories, particularly chromite, resulting in substantial growth and loss of strength. It has, for example, been observed that the roof of an all-basic furnace may become rough even before steelmaking commences, particularly at points where alternating, reducing and oxidising conditions might be expected. In the new processes attempts are made to control furnace atmosphere, but the high evolution of carbon monoxide frequently results in strongly reducing conditions, followed later by an oxidising atmosphere.

## OPEN-HEARTH FURNACES

### WITH OXYGEN FOR COMBUSTION

Oxygenation is particularly useful where limitations on air input, waste gas removal, or checker chamber size, prevent an increase in fuel rate. A typical rate of oxygen feed for a 100-ton furnace would be 20,000–40,000 cu. ft. per hour. Adequate charging facilities are of course essential, since the potential gain is mainly in charge to melt time.

(a) CONSTRUCTION

Appreciable gains—of the order of half an hour in tap to tap time—can be achieved by introducing oxygen into the air entering the checkers. The more usual procedure, however, is to inject the oxygen either through a separate hole in a fuel burner, or through a second lance parallel to and generally below the fuel stream. Considerable study has been made, *e.g.*, by the International Flame Research Foundation at IJmuiden, on the optimum separation between the fuel and oxygen jets. If the distance is too great the procedure is more akin to jetting than oxygenated combustion. In general the oxygen stream is introduced a few inches to a foot below the fuel stream, it being thought that this provides maximum heat transfer to the charge with minimum additional heat transfer to the roof.

More dramatic is the effect of using an oxygen-fuel lance, introduced through the roof, a method tried out quite extensively in the United States and France. The burners used may be designed for the sole purpose of introducing oxygenated fuel, or may be suitable also for oxygen jetting during refining. Preliminary experience suggests that

the former is more desirable and that substantial gains can be obtained, particularly where the burners can be moved to provide an optimum distance between burner tip and melting scrap. Attempts have been made, notably in Germany and Russia, to design furnaces in which the fuel is burned with oxygen alone. Dramatic increases in output have resulted but the current opinion would appear to be that the refractory wear is too severe to permit of economic operation.

#### (b) MATERIALS

Where oxygen is used to assist combustion of normal fuel quantities, and roof temperature is adequately controlled, no appreciable increase in refractory wear should result. In general, however, oxygen is used to enable increased amounts of fuel to be burned. Silica roofs can be given some protection from the more severe conditions by increasing the distance between the roof centre and the sill, but the stage is soon reached at which basic roofs become essential, if only because silica roofs are likely to be melted by flames deflected from high piled scrap. If deflection is avoided by keeping charging rates low, the advantages of using oxygen largely disappear. Although the all-basic furnace was developed before cheap oxygen became available, there is little doubt that it was the availability of oxygen at a few shillings per thousand cubic feet that led to the recent swing to all-basic construction—particularly in the United States and Great Britain. The suddenness of the swing is illustrated by the fact that there were only about 10 all-basic furnaces in Great Britain during the period 1948–58, compared with nearer 70 in 1960.

#### (c) LIFE AND CAUSES OF FAILURE

Under certain conditions, *e.g.*, with furnaces burning tar or pitchcreosote, oxygen may even lead to increased roof life by decreasing the amount of fume formed. Generally, however, faster driving, including both oxygen and higher fuel rates, leads to a reduction in roof life even on all-basic furnaces.

It also results in heavier wear in downtakes and checkers. The mechanism of roof wear is still far from clear, roof temperatures being well below the melting point of basic roofs. All that can safely be said is that faster driving generally means higher temperatures and higher iron oxide concentrations, which in turn mean more rapid roof failure, though not necessarily higher refractories costs. The latter must in any case be always considered in conjunction with output gains and changes in fuel consumption.

#### (d) LINES OF IMPROVEMENT

If furnaces are to be operated continuously at high fuel rates and with part of the fuel introduced through roof burners, then major advantages in design may well prove desirable. It is unlikely, for example, that the present rectangular shape, which originated from the introduction of all the fuel through the furnace ends, will prove to be ideal. Increased oxygen usage may also lead to smaller checkers and flues, and even, in extreme cases, to the complete elimination of regeneration. Here the gain in refractories must be offset by reduction in fuel efficiency. Such design changes may, however, prove small compared with those ultimately demanded by oxygen lancing and jetting.

### OPEN-HEARTH FURNACES

#### WITH OXYGEN LANCING OR JETTING

The first trials of blowing oxygen into open-hearth furnace baths through steel pipes, were, to say the least, rather frightening, it being thought that such procedures might well result in the destruction not only of the furnace but also of the operator. Those daring to insert two such lances simultaneously were considered extremely bold, yet today in Japan, as many as ten lances are introduced at one time and blowing commenced at far higher levels of carbon in the bath than was previously considered safe, either on grounds of excessive boiling (*see* fig. A3, p. 714, for modest boil) or excessive fuming.

#### (a) CONSTRUCTION

Lancing by means of 1 in. diameter uncooled steel pipes is still common but there is an increasing tendency, particularly in the United States, to use roof jets, of the type developed by the Steel Company of Wales. These are heavily water-cooled, are normally copper tipped, and may be of the original single-hole or the multiple hole (usually six) type. Blowing rates vary greatly but are typically 30,000 cu. ft. per hour for a dry lance and 70,000 cu. ft. for a roof jet.

Far higher blowing rates have, however, been used in the 500-ton open-hearth furnace at the Steel Company of Canada, described by A. K. Moore, at the 1962 A.I.M.E. Open-Hearth Conference. This furnace broke all existing records by doing a whole campaign with an average output of over 100 tons per hour. Its general design was conventional, it being a 7-door furnace (*see* fig. A4, p. 719), fitted with a roof of the box-car type, single uptakes, and 2-pass regenerators. The vastness of the flue system can be gathered from the fact that the induced fan had a capacity of 1500 horse-power. The roof itself was of the suspended/hold down type, being 15 in. thick and without ribs. The



*Fig. A4. Record breaking seven-door 500 ton open-hearth furnace at Steel Company of Canada.*



*Fig. A5. Microradiograph of iron rich zone of used chrome-magnesite brick (x28) showing direct bonding between chrome grain and highly crystalline magnesia.*

arch was extremely high, the distance between sill and roof centre being 13 ft. or more. It operated on approximately 50 per cent. hot metal and 50 per cent. scrap, the latter being charged in boxes of 60 cu. ft. capacity and at such a rate that the charging time was reduced to only 45 minutes. The furnace, after several campaigns, had a roof life of the order of 400 heats. Its maximum firing rate was 400,000,000 B.Th.U. per hour, while the maximum oxygen flow through the three roof lances reached the record figure of 480,000 cu. ft. per hour—about  $\frac{1}{2}$  ton per minute. It is hardly surprising that resort was made to substantial water-cooling of sidewalls and that water sprays were injected into the gases entering the checkers in order to achieve a reasonable operating temperature. It is most encouraging that under such conditions the refractory consumption was only about 16 lb. per ton; including material used for spraying but not of course fettling. The fuel consumption was approximately half the normal figure, *viz.*, 2.1 million B.Th.U. per ton, while oxygen consumption was given as 1175 cu. ft. per ton.

(b) MATERIALS

Dry lancing has often been carried out with silica roofs, but where high inputs of oxygen are used, either through lance or roof jets, the tendency is to swing almost immediately to all-basic construction. Many users have also discovered that under conditions where slag splashing is substantial, a change from chrome-magnesite to magnesite-chrome bricks gives improved life, due presumably to the greater slag resistance of magnesia.

(c) LIFE AND CAUSES OF FAILURE

Observation of furnaces during lancing or jetting shows both substantial impact of splashed droplets on the roof and excessive iron oxide fumes. In general there is a clear layer, probably containing iron vapour, of a foot or so between the bath and the fume. Above this the fume is so dense that viewing of the roof, either by eye or pyrometer, is no longer effective. In the Japanese furnaces, referred to above, the change from no oxygen usage to oxygen for combustion and introduction through seven lances, led to a doubling of output, tap to tap times in a 140-ton furnace being reduced to less than four hours, but also to a substantial reduction in roof life. Oxygen consumption on these furnaces was high—1500 cu. ft. per ton—but the marked drop in fuel consumption, and the greater outputs, were said to more than compensate for the cost of oxygen and the reduced furnace life. The lives obtained on all-basic furnaces vary over an extremely wide range but



in general lie between  $1\frac{1}{2}$  and 4 times those obtained on silica furnaces operated without oxygen. Whether all-basic construction is economic at a given roof life can only be determined by an overall cost balance at the particular plant concerned.

(d) LINES OF IMPROVEMENT

Hydrodynamic studies show that splashing can be greatly reduced for example by the use of multiple jets. Frequently, however, improved roof life may be associated with lower sidewall life, the droplets merely tending to move sideways rather than upwards. Under these conditions magnesite bricks may well give better results in sidewalls than the traditional chrome-magnesite lining. The present trend towards high magnesite content in roof bricks is likely to continue, and it is quite possible that a straight non-spall magnesite may be produced that will give better service in roofs than bricks containing chrome ore. It has also been shown that under the severe conditions existing in Ajax furnaces—see later—a checkerwork of magnesite and chrome-magnesite bricks behaves very satisfactorily. Another, and valuable, line of development, still at an early stage, is the direct bonded chrome-magnesite brick, in which intercrystallisation between magnesite and chrome is achieved by keeping the silica low and employing unusually high firing temperatures (*see* fig. A5, p. 720). Increasing use is also likely to be made of spraying. Given improvements both in the guns and cements used this practice may well prove economical as a way of balancing roof life with that of other furnace parts.

## AJAX FURNACES

Experimental work on the use of oxygen in large tilting furnaces started at the Appleby-Frodingham Steel Company in 1949. It was not, however, until 1955, when basic roofs were introduced, that rapid progress was made, and not until 1957 that the first conversion of a tilting furnace to an Ajax furnace commenced. It will be seen from the following description that the Ajax furnace is a sort of half-way house between the LD or Kaldo and the open-hearth furnace. Since the LD is itself likely to undergo changes as it grows larger, and more experience is obtained, including possibly such additions as lids and heat regenerators, the Ajax, in addition to being economic, may indicate something of future trends.

The particular units described, operate on a high metallurgical load (1.1 per cent. phosphorus) and use up to 1400 cu. ft. per ton of oxygen at an input rate of 55,000–75,000 cu. ft. per hour. The fuel consumption is very low (9.5 therms per ton), *i.e.*, about a quarter of that employed



*Fig. A6. Ajax furnace at the Appleby-Frodingham Steel Co.*



in ordinary hot metal units. Fig. A6, p. 723, shows a photograph of an Ajax furnace, and fig. A7, p. 726, a general outline, from which it will be seen that the usual end burners have been replaced by 30 ft. long water-cooled lances, whose tips reach roughly to the bath level. Small burners equipped to burn coke-oven gas and oxygen are fixed in the furnace corners. The downtakes, slag chambers, and regenerators are encased in cylindrical air-tight steel shells and are coupled to the furnace hearth and the waste-heat boiler by refractory-lined steel pipes. The whole system, from the furnace laboratory to the flues, is insulated with  $4\frac{1}{2}$  in. high-quality insulating brick and 1 in. magnesia asbestos against the shell.

The advantages of such furnaces may be summarised as follows:

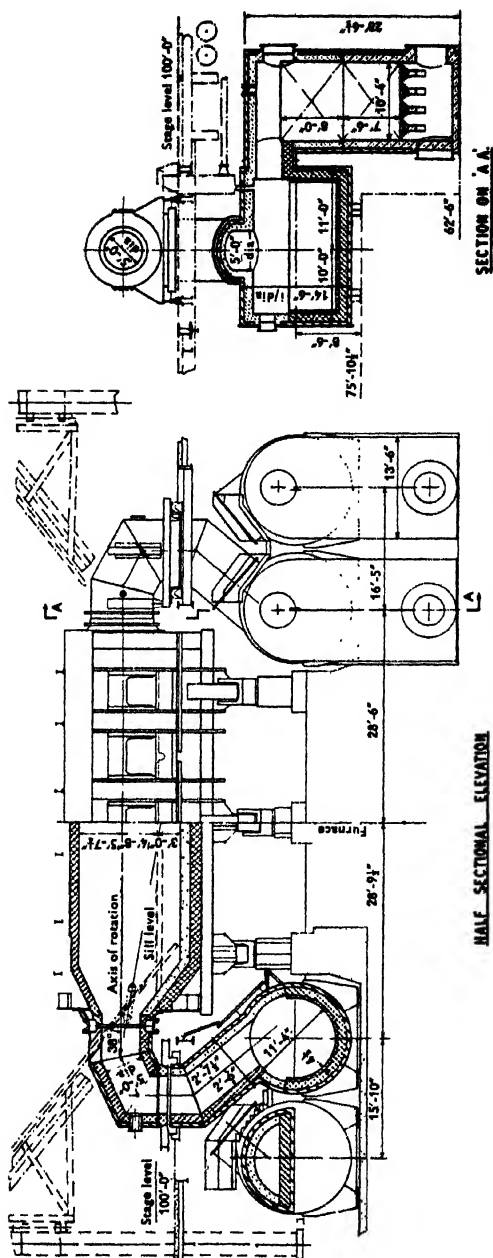
- (a) Increased thermal efficiency due to reduced air infiltration and insulation.
- (b) Reduced wastage of refractories in "dead" spaces.
- (c) Slag pockets, regenerators, and flues, structurally self-supporting.
- (d) Low infiltration, resulting in greater steam production at the waste-heat boiler and permitting a smaller cleaning plant.

The duplication and interchangeability of slag pockets, regenerator chambers, uptakes, and port ends, have resulted in 99 per cent. availability below stage, the campaign life being dictated by the roof life.

#### ROOF

Prior experience showed that silica roofs would be hopeless on the Ajax furnace—even basic roofs requiring patching above the lance after only a few days' operation. Roof-temperature measurements, made by means of rare metal thermocouples, indicated temperatures of the order of  $1900^{\circ}\text{C}$ . By reducing oxygen flow rate, and the use of multi-hole lances, the life of a chrome-magnesite roof was increased to about six weeks. Further improvements followed an increase in roof rise from 2 ft. 6 in. to 4 ft. 0 in. and a decrease in roof radius. These, together with a checkerboard construction, using alternate magnesite and chrome-magnesite bricks, separated by steel sheets, have resulted in lives of the order of 12 weeks. The results of trials of numerous different roof bricks may be summarised as follows: the best lives with chrome-magnesite have been given with bricks of particularly low silica content and high strength in torsion at  $1300^{\circ}\text{C}$ . Typical properties of the bricks giving good service are as follows:

Apparent porosity,	per cent.	..	20.0 to 23.0
Twist in torsion, 1 hr. $1300^{\circ}\text{C}$ .		..	7.0 to 17.0
(radians $\times 10^3$ )			



HALF SECTIONAL ELEVATION

Fig. A7. General construction of Ajax furnace.

## Permanent volume change—

2 hr. 1700°C.	per cent. . .	less than 1.0
SiO <sub>2</sub>	per cent. . .	3.3 to 3.7
Cr <sub>2</sub> O <sub>3</sub>	per cent. . .	22.0 to 25.0
MgO	per cent. . .	43.0 to 53.0

Consumption of roof bricks at present amounts to approximately 7 lb. per ton of steel.

The mechanism of failure is still far from clear and appears to have changed over the years from the slabbing typical of earlier operation to a type of wear more akin to slag attack. Examination of used bricks shows iron oxide and lime to be highest at the working face and silica to be low but increasing to a peak value at some distance in. Further, it would appear that the bricks are mechanically weak on the hot side of this silica rich zone. When the hot side is lost much of the lime appears to be lost with it, while the silica moves inwards, its concentration gradually increasing as the roof gets thinner. The Ajax roofs described have been built with a rigid (hold-down) construction. Trials of other constructions, *e.g.*, the so-called "Continental", might yield still longer life by enabling roofs to be run thinner without risk of collapse.

## FRONT AND BACK LININGS

On the first furnaces these were mainly chrome-magnesite but with the present-day furnaces the best results are obtained with fired or unfired (metal-case) magnesite bricks, probably because of their greater resistance to splashed slag.

## BANKS AND BOTTOM

A magnesite hearth rammed over a magnesite brick bottom but with tarred dolomite banks is now standard. Fettling consumption is quite substantial, some 65 lb. of burnt dolomite per ton being used. If, however, allowance is made for the lime that would otherwise be needed the net consumption is nearer 40 lb.

## PORT ENDS

In the earlier furnaces these were built of magnesite-chrome bricks but it now appears that 73 per cent. alumina brick give a better performance and lower operating costs.

## UPTAKES

These also were originally made of fired magnesite-chrome brick, but silica bricks are now found to be quite satisfactory in the upper section.

## SLAG CHAMBERS

These were originally basic but are now found to be quite satisfactory in silica brick.

## CHECKERS

It was thought that basic bricks would be essential due to the high temperatures and high iron oxide concentration, in fact 42 per cent. alumina brick have proved adequate. The checker chambers are constructed in silica brick.

## REFRACTORY CONSUMPTION AND COSTS

The savings in refractories, due to the adoption of the Ajax process, are illustrated by the following table taken from Jackson's 1962 paper in *Steel & Coal*:

TABLE AII

<i>Bricks in lb./ton ingots</i>	<i>Standard open-hearth (1,000,000 tons)</i>	<i>Ajax avg. (1,000,000 tons)</i>	<i>Best campaign to date (100,000 tons)</i>
Silica ..	14.8	2.1	1.3
Firebrick ..	5.8	1.5	0.9
Magnesite ..	1.9	4.7	6.0
Chrome-magnesite..	11.1	13.5	6.1
	33.6	21.8	14.3

It will be seen that silica and firebrick consumptions, once major items, are now almost negligible, but that the usage of magnesite and chrome-magnesite is still substantial.

Running costs in 1961 for the various parts of an Ajax furnace were given as follows:

Roof .. .. .	2s. 4d.
Front and back walls .. ..	1s. 10d.
Bottoms and banks .. ..	2d.
Ports .. .. .	8d.
Uptakes .. .. .	7d.
Slag chambers .. .. .	9d.
Checker filling and chambers ..	2d.
Flues .. .. .	1d.

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6s. 7d.

It will be seen that both the poundage and the costs are appreciably lower than with conventional furnaces, in spite of the fact that driving rate has been roughly doubled.

## STEAM-OXYGEN CONVERTERS

A number of plants on the Continent and a few large plants in this country, notably Richard Thomas & Baldwins Ltd., and the Steel Company of Wales, have operated steam-oxygen converters, a principal objective being the production of low nitrogen steel for deep drawing. The obvious way of speeding up the basic Bessemer process, *i.e.*, the replacement of air by oxygen, has unfortunately a disastrous effect on bottom life. A number of alternatives, in particular diluting the oxygen with steam or carbon dioxide, have, however, been tried, and of these steam-oxygen appears to be the most acceptable. Preliminary experience at the Steel Company of Wales has been summarised by Harrison and Truscott and later by Boddye and Elderfield in their Iron and Steel Institute Special Report No. 74 paper.

### (a) CONSTRUCTION

As with the Bessemer process the refractory problems fall naturally into two groups, *viz.*, side linings and bottom. The former may be rammed or built with blocks, while the latter is always produced as a separate monolith. The manufacturing procedure is similar to that of the Bessemer process, except that extra care must be taken to avoid hydration of the bottom by the steam used to dilute the oxygen. The difficulty has been largely overcome by the ingenious use of 12 mm. i.d. copper pipes as linings to the tuyere holes. These are sealed into the bottom with air setting cement. Lining dimensions are similar to those of basic Bessemer converters, the thicknesses in the 50-ton vessel at the Steel Company of Wales varying from about 3 ft. 6 in. at the base to 1 ft. at the mouth. The bottom itself is cylindrical, being roughly 10 ft. in diameter, 3 ft. thick and containing 282 tuyere holes, each lined with its copper pipe.

### (b) MATERIALS

The quality of the dolomite used in side-walls and bottoms has been shown to have a remarkable effect on life. Boddye and Elderfield, for example, showed increases in bottom life from 30 to 40 charges as a result of an increase in bulk density from 2.6 to 2.9 g.p.ml. Control of grading and tar quality and quantity are also of the highest importance. Nearly 200 tons of dolomite are used, the ramming mix being made by charging the mill with dolomite having the following grading:



mm.	lb.	(kg.)
4-8	924	(420)
2-4	462	(210)
0-2	924	(420)
0-0.5	770	(350)
Total	3080	(1400)

To this batch is added 7-8 per cent. of tar and the whole milled for approximately 20 minutes. The dolomite is rammed with  $\frac{1}{8}$ -in. wire mesh behind the shuttering to prevent loss of material during stripping. Heating-up is done by means of a coke-oven gas flare, a burning in period of 6-8 hours being normal. The total time required for a reline is approximately 4 days.

The procedure used in preparing the batch required for ramming bottoms is described as follows:

- (i) 1760 lb. (800 kg.) of >8 mm. material is fed into the pan mill and crushed for about 15 minutes.
- (ii) 18-21 gal. of tar are added and mixing is continued for a further period of 20 minutes.
- (iii) 2-3 gal. of tar are then added followed by the 440 lb. (200 kg.) of 2-4 mm. material, mixing is continued for 10 minutes.
- (iv) 2-3 gal. of tar are again added followed by the 220 lb. (100 kg.) of 0-2 mm. and the 176 lb. (80 kg.) of 0-0.5 mm. doloma. The final mixing time is 15 minutes and this completes the operation.

The total mixing time is 50 minutes and the amount of tar used about 11 per cent. Forming is carried out by means of the usual vibrating table, the temperature of the mix in the mould being kept at 70-75°C. by means of flares. The rammed bottom is then fired by blast-furnace gas in a special oven, the temperature being raised slowly to 600°C., held for 72 hours and then gradually reduced. The complete cycle takes 15 days, and the bottom so produced can be stored for 5-6 weeks before use.

#### (c) LIFE AND CAUSES OF FAILURE

Side wall life in these steam-oxygen converters is approximately 240 heats, but bottom life only 40-50 heats, the published record being 60 heats. According to certain workers, *e.g.*, Klarding, the wear on the side walls is proportional to the thickness of the decarburised zone formed behind the slagged surface. Care should, therefore, be taken to minimise atmospheric oxidation. Silicon content of the metal, which Bell showed was highly important with basic Bessemer linings, does not appear to be an important factor with these steam-oxygen converters,



*Fig. A8. LD vessel being charged with molten iron.  
(Courtesy Richard Thomas & Baldwins, Spencer Works).*



which, it is suggested, are first weakened by carbon loss and then fail by physical abrasion and chemical attack.

(d) LINES OF IMPROVEMENT

The relative simplicity of the LD vessel which can also be used to make deep drawing steel makes unlikely any extension of the steam-oxygen process. The linings in existing units will doubtless be improved with additional experience, the factors involved being similar to those described later in connection with the lining of LD, Kaldo and Rotor vessels.

BASIC OXYGEN CONVERTERS  
(LD, LDAC, AND OLP)

On 27 November 1952, the first LD (Linz-Donawitz), or BOF (Basic Oxygen Furnace), shop went into operation at Linz under the direction of Vereinigte Österreichische Eisen und Stahlwerke. By May 1962, some 8-million tons of steel had been made by this process at Linz and some 16-million tons of annual capacity installed throughout the world (*see* fig. A8, p. 731). With the plant now under construction a figure of 30-million tons should soon be reached, while 70-million has been given as the estimate for 1965. The impact of this new process is most dramatically shown in relation to Japan, where the expansion of steelmaking capacity is rapid and the new units almost entirely of the LD type. Some 20 vessels are already in operation, varying in size from 30 to 70-ton capacity, and many others are scheduled. Nagai estimates that by 1962 some 9-million tons per annum of LD steel will be made in Japan compared with 16-million in open-hearth and 6-million in electric furnaces. By 1970 a total of 48-million tons is expected, of which 25-million will be LD, 15-million open-hearth and electric steel approximately 8-million. Although development elsewhere is likely to be less rapid, substantial installations have already been made in Belgium, France, Germany, Great Britain and Italy, as well as in the United States, Australia and Brazil. Canada was early in the field, having two plants by 1958, while one of the new Indian plants—Rourkela—also operates the LD process.

(a) CONSTRUCTION

Most of the LD vessels built so far strongly resemble basic Bessemer units and, indeed, certain of them are in fact conversions, the tuyere section having been rammed solid and a single oxygen lance introduced from above. The larger vessels appear for the most part to have a greater breadth to height ratio, while Weitzer and Rinesch predict a

change to symmetrical vessels, probably with some type of lid or roof. A notable improvement was the introduction of a taphole in the side of the vessel: this enables slag to be held back but brings with it new refractory problems. The amount of oxygen blown through the water-cooled lance varies from 4000 cu. ft. per minute for the 30-ton Austrian LD units to 13,000 to 20,000 cu. ft. per minute on the 200-ton basic oxygen furnace at Jones and Laughlin in Cleveland. The consumption of oxygen varies greatly with metallurgical load, notably phosphorus and silicon, but is of the order of 1500 to 2000 cu. ft. per ton.

LD plants operate with relatively low phosphorus irons but it has been shown, particularly by the Belgians (LDAC process) and the French (OLP process) that high phosphorus irons can be handled in such vessels given lime injection and double slags. The vessels used in the OLP process, *e.g.* the 50-ton converters at Denain and 30-ton converters at Dillingen, also show a lower height to diameter ratio. Details of these vessels and of the preliminary results obtained with them were reported to the Iron and Steel Institute's Symposium by Français, Bauer, and Vayssière. They state that the shape was the result both of practical experience and model studies by Leroy, Chedaille and Mathieu. Mouth size is considered particularly important, being kept as small as possible in order to reduce heat losses. It is suggested that an increase in mouth diameter on a 50-ton converter from 4 ft. 3 in. to 6 ft. 6 in. (1.3 to 2 metres) reduces by 20 per cent. the amount of cold scrap that can be melted. Bath depth is also considered important, a figure of 3 ft. 11 in. (1.2 metres) being given for a 50-ton and 5 ft. 2 in. (1.6 metres) for a 100-ton vessel. The importance of adequate lining life is brought out by the substantial repair time, stated to be about three days.

The first LDAC vessel in Great Britain, installed at the Ebbw Vale plant of Richard Thomas & Baldwins Ltd., has been described in the Iron and Steel Institute's Symposium by Carr. It has a 25-ft. high shell of 14-ft. diameter, and is a conversion from an existing Bessemer unit. Its internal volume varies greatly—from 1000 to 2000 cu. ft.—during a campaign. The bottom of this 30-ton vessel is removable—another result of its origin.

#### (b) MATERIALS

The general arrangement of the lining in the 50-ton Linz unit is shown in fig. A9, p. 735, together with the typical wear contour. It will be seen that the lining consists of three layers, an outer safety course, an inside brick or rammed lining and a rammed layer between the inner and outer linings. Tarred dolomite in block form is the material

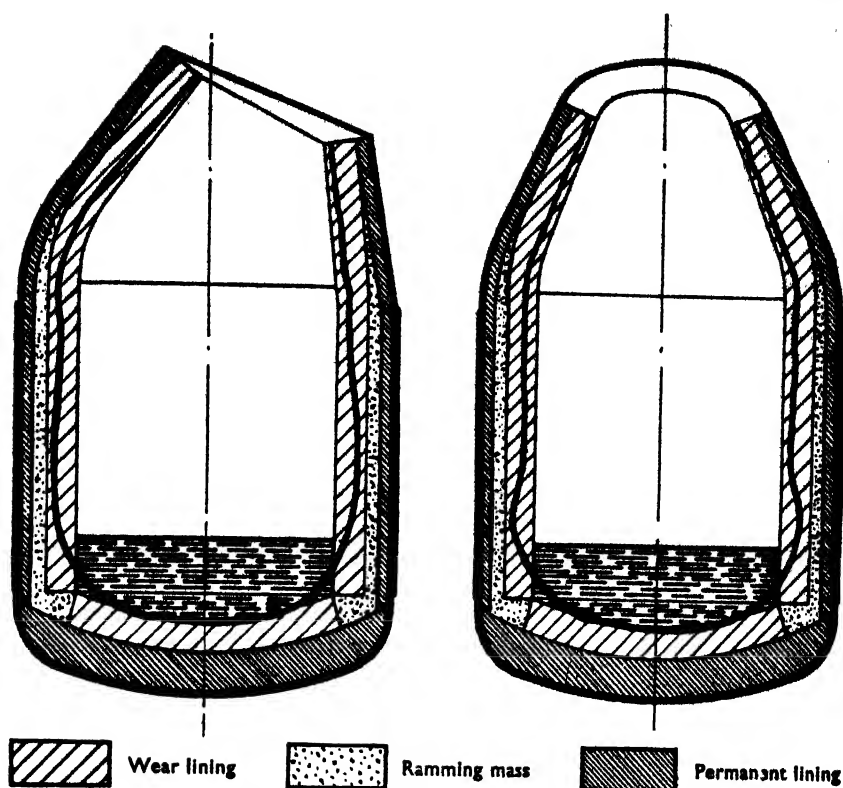


Fig. Ag. General construction and wear lines on LD vessel at Voest. (After Weitzer and Rinesch.)

most commonly used in LD vessels, but the present linings at Linz are made from tar-bonded high-lime magnesite blocks. This is a natural material, whose composition after dead-burning is roughly as follows:

MgO	..	65-80
CaO	..	10-25
SiO <sub>2</sub>	..	2-5
Al <sub>2</sub> O <sub>3</sub>	..	about 1
Fe <sub>2</sub> O <sub>3</sub>	..	4-6
Ignition loss	..	0.5-2

The approximate grading is given as 60 per cent. coarse, 40 per cent. fine with a tar addition of 4-6 per cent. The latter must be free from

water and naphthalene but contain small quantities of light oils and about 70 per cent. of pitch.

By contrast the linings at the Tobata plant of the Yawata Iron and Steel Company Ltd. (*see* fig. A10, below) have an inner top lining of

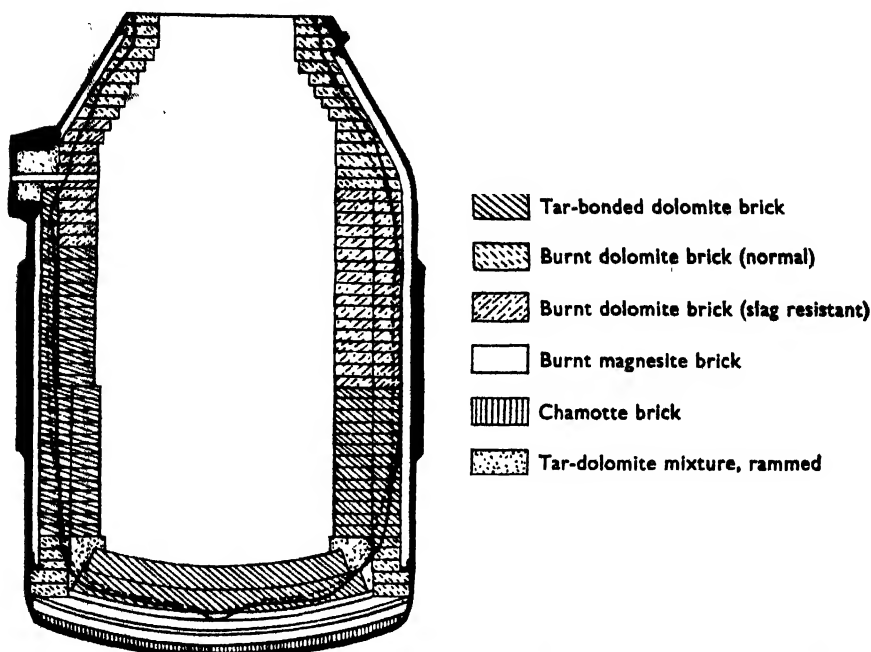


Fig. A10. Brickwork and wear lines on LD converter at Tobata, after 420 heats. (After Ohba, Ikenoue, and Nishikawa.)

stabilised dolomite bricks and an inner bottom lining of tarred dolomite blocks. It is suggested that the stabilised dolomite bricks give better service in this position because of their greater mechanical strength and consequently better resistance to abrasion, *e.g.* by scrap. The properties of these stabilised bricks are similar to those given for this type of brick in Chapter IV of the present text. The tarred dolomite bricks have of course quite different properties, their porosity being as low as 7.5 per cent. (due in part to the tar), their cold crushing strength 4900 lb. per sq. in. (350 kg. per sq. cm.) and their fail point in the refractoriness-under-load test (28 lb. per sq. in. – 2 kg. per sq. cm.) 1710°C. Their silica content is low, being about 3 per cent. compared with 10 per cent. for the stabilised material. Magnesite bricks made from iron bonded clinker and having good volume stability are used in the permanent lining.

Relatively little has, as yet, been published on the types of brick used in these vessels in the United States and Canada, though these can be divided roughly into three main groups, *viz.*, magnesite, tarred dolomite, and tarred dolomite with the fines replaced by magnesite. The object of this latter procedure is mainly to reduce hydration and thereby increase "shelf life" but it should also increase slag resistance. In the preliminary experiments at Ebbw Vale, 14 in. long tarred dolomite blocks were used for the working lining and tarred dolomite ramming between this and the safety course. The taphole was formed of magnesite blocks and had an internal diameter of 3 in. It is surprising to note that even this relatively small (30-ton) vessel requires over 100 tons of lining material. Burning in procedure varies considerably, a typical time at the Ebbw Vale plant being 16–20 hours. The temperature required is usually achieved by either air or oxygen being blown on to a bed of coke. Given oxygen satisfactory temperatures can be obtained in six hours or less without disturbance of the lining. At Dillingen gas heating is used. It is slower than coke but considered safer. With the OLP process at Denain the lining is constructed of approximately 125 tons of tarred dolomite blocks and some 40 tons of tarred dolomite ramming material. Magnesite bricks are again used for the taphole. Trials have also been made of straight magnesite bricks and it would appear that their use may be justified in certain positions, *e.g.* the nose lining.

Nagai suggests that improved results might be obtained with a special clinker formed from a mixture of sea-water magnesium hydroxide and slaked dolomite, pelletised and clinkered in a kiln. Numerous different mixtures have been tried, both with and without mineralisers, such as iron oxide and boric acid. This magnesia-dolomite clinker has excellent properties and in particular yields a product with a high refractoriness-under-load. Mineralogically it is a mixture of magnesia (55–62 per cent.), lime (30–35 per cent.), and small amounts of dicalcium ferrite and brownmillerite ( $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ ). Boric acid additions, which yield magnesium borate, are stated to have a remarkable effect on the mineralisation of periclase.

#### (c) LIFE AND CAUSES OF FAILURE

The life obtained varies greatly with the size of unit and the metallurgical load. It was hoped that lime injection, as used in the OLP and LDAC processes, would reduce refractory wear by reacting with iron oxide and silica at the point of formation. Such reactions doubtless occur but as the use of lime injection is normally limited to high phosphorus irons, which yield a high slag bulk, the net wear is usually



greater than with normal LD operation. As in the Thomas process, high silicon iron tends to cause more wear, but manganese has little effect. Heavy scrap can also do damage if not charged on to a bed of light scrap or lime. A factor considered particularly important is lance position, vertical and central impingement on the bath being considered vital, even a slight deviation leading to excessive wear on one side of the lining. In the 50-ton vessels at Linz, wear is stated to be worst in an area opposite the trunnions, i.e., in a region about 1 ft. 8 in. ( $\frac{1}{2}$  metre) below to 3 ft. 3 in. (1 metre) above the bath surface. Wear on the bottom is negligible and removable bottoms, such as are used in the Thomas process, are not therefore necessary. Adequate space above the bath is considered essential to avoid heavy slopping, with its serious effect on the bricks in the hood and mouth.

The consumption of magnesite at Linz is low, viz., 8.8 lb. (4 kg.) per ton of steel, and lives of 380 heats are obtained with 30-ton converters and 400 heats from the 50-ton converter. The record life so far reported is 527 heats. These lives correspond to wear rates of about 1–2 mm. per heat, with the best as low as 0.8 mm. Tests have been made at Linz using dolomite from various countries, dolomite-magnesite mixtures, and fired magnesite bricks of both low and high iron oxide content. The general conclusion, both here and elsewhere, would seem to be that fired magnesite bricks though giving the longest life are not the most economic.

Lining lives obtained in Japan are good, a figure of 400 heats or 11 lb. (5 kg.) per ton being given for the 60-ton converter at Tobata. The even wear contour shown in fig. A10, p. 736, suggests that careful choice of materials has led to efficient use of the various brick qualities. Comparatively little information is available regarding lining lives in the United States and Canada, but the levels generally would appear to be somewhat lower, Glasgow, for example, reporting figures from 98 to 167 heats for the early campaigns on the 200-ton unit at Cleveland, and McMulkin reporting a life of 260 heats on tarred dolomite linings at Dofasco. According to Français *et al.*, an average life of 157 heats was obtained on 18 OLP campaigns at Denain with a refractories consumption of 31.2 lb. (14.2 kg.) per ton. Off-centre lances, too big a distance between lance and bath, and high iron oxide in the slag, were all found to increase wear.

A fairly detailed study has been made at Yawata, and certain other steel and refractory companies, of the causes of wear in LD converters. It would appear, for example, to be widely agreed that dolomite bricks, either tar-bonded or stabilised, give service comparable with the more

expensive fired and unfired magnesite bricks. Further that with unfired materials slag penetration is limited to a narrow surface zone, whereas with fired products slag penetration is deeper. Evidence is accumulating that carbon formed from tar plays a vital role, it having been noticed that attack is preceded by decarburisation of the surface layers. It has been suggested, *e.g.*, by the author, that carbon protects the lime in the dolomite by converting the attacking iron oxides to FeO or Fe. The latter has little or no corrosive action, while FeO, though highly soluble in magnesia, has only limited solubility in lime. This view is supported by evidence from at least two laboratories, where petrological examination and X-radiographs have shown the presence of numerous globules of metallic iron in a region immediately behind the decarburised zone. If the postulated mechanism is correct then considerable research would be warranted both on achieving improved impregnation with carbon and on maintaining this carbon in service. It suggests, for example, that care should be taken to avoid strongly oxidising atmospheres both in burning-in and use of the lining. The increase in LDAC lining life quoted by Carr from 80 to 180 heats, as a result of decreasing time between blows (fig. A11, below) may well be explainable in terms of

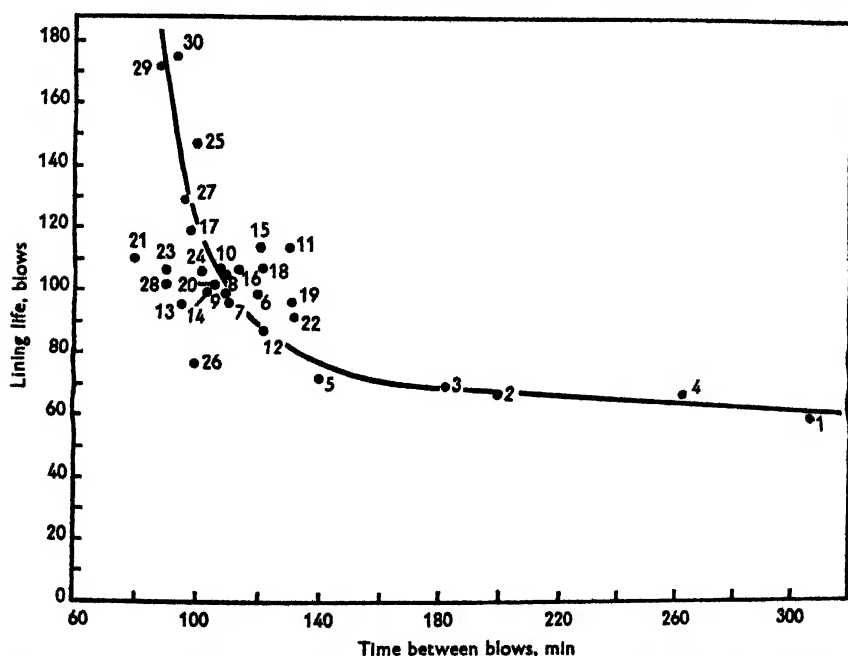


Fig. A11. Lining life on LDAC converter as function of time between blows. (After Carr.)

carbon loss during standing. Many other factors of course affect lining life: it has, for example, been shown on the LDAC vessel at Ebbw Vale that the wear pattern is a function of the rest positions, due apparently to slag line cut, also that the bulk density of the dolomite has a marked effect on durability.

(d) LINES OF IMPROVEMENT

There would appear to be room for improvement in each of the three main fields, *viz.*, construction, materials, and operation. On the design side the reference in the bibliography to hydrodynamic studies, *e.g.*, those of Kootz (*see* fig. A12, below), Holmes and Thring, Holden and

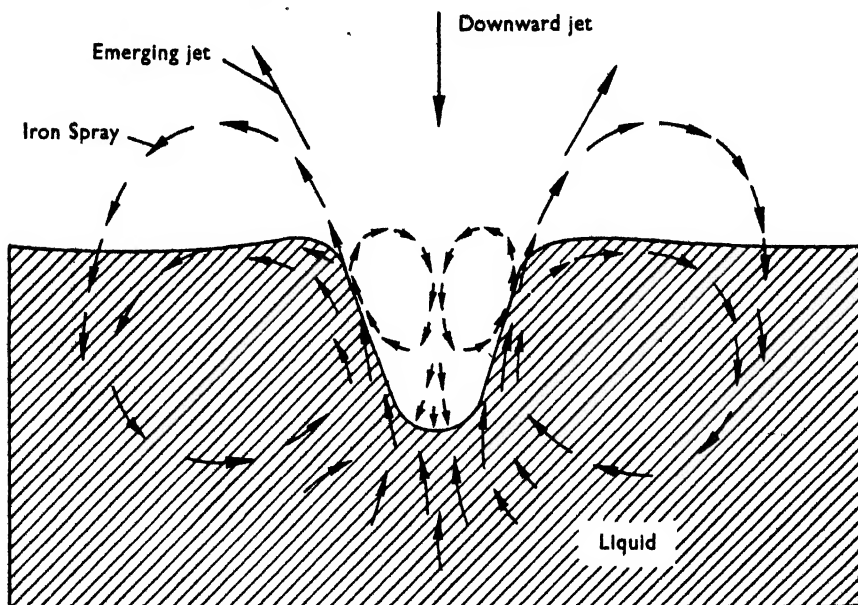


Fig. A12. Flow patterns in bath and gas when blowing increased until spraying occurs. (After Kootz.)

Hogg, and Kun Li, show that much remains to be done regarding movement of fluids both within the bath and in the gases above. To this must be added a study of splashing, which reaches a high level and depends among other things on the relatively unknown drop dynamics. The vast quantities of fume must also be responsible for corrosion, though they may also tend to hold down lining temperatures by acting as a radiation shield. As LD vessels increase in size there may be a special case, as with the open-hearth furnace, for the use of



*Fig. A13. Kaldø vessel at Domnarvret, Sweden.*



several lances and of lances of the multi-jet type. There would also seem to be a real need for some method of mouth closure, though the difficulties involved can readily be envisaged by anyone who has witnessed a really wild heat on an LD converter.

If it is true that carbon protects the dolomite by keeping iron in a reduced condition, there would seem to be a need for the development of special clinkers, such as the magnesite-dolomite batches described by Nagai. Given high refractoriness and the associated high porosity such clinkers could be impregnated with carbon prior to mixing with the finer fractions and tar in the brickmaking. Recent progress in direct bonding of chrome-magnesite bricks suggests that straight magnesite bricks, not dependent on a silicate bond, may yet be developed that have sufficiently high spalling resistance to enable them to prove economic in competition with tarred dolomite.

On the operating side tighter control of atmosphere during burning in and when standing, such as might be achieved by the use of lids and protective atmospheres, may well prove worth while. Finally, an obvious answer to shorter lining life, *viz.*, efficient fettling between heats, would still appear difficult in spite of the good results obtained by Davies with powdered lime on the refining vessel at Brymbo and the frequent suggestion of oxygen-fuel-refractory powder type burners.

## ROTATING VESSELS

Two important processes, *viz.*, the Kaldo and Rotor, produce steel from molten iron, scrap, and iron ore, in a rotating drum. As will be seen from the following description, such rotation has advantages but also introduces new problems. Comparison of refractory performance with the LD is difficult, in that the rotating vessels are generally used with iron high in phosphorus compared with that employed in the normal LD. and permit the burning of carbon monoxide within the vessel. All the evidence, however, suggests that both refractory wear and the relining problem are more serious than with fixed vessels.

## KALDO

### (a) CONSTRUCTION

The first 30-ton Kaldo vessel was operated at Stora Kopparbergs, Domnarvet, Sweden, in May 1956, having been designed on the basis of experience gained on a 3-ton pilot unit. As will be seen from figs. A13, p. 741 and A14, p. 744 the oxygen is introduced through the mouth of the vessel, which also serves as a waste-gas outlet. Because rotation provides ample turbulence only low pressure oxygen need be employed. Cooling of the bath can be achieved by the addition either of iron ore

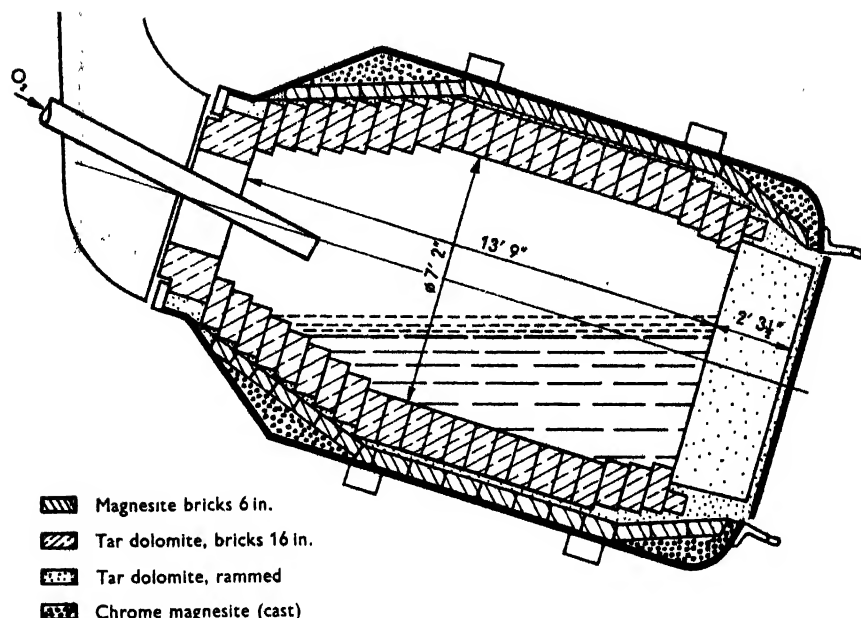


Fig. A14. 30 ton Kaldo vessel at Domnarvet showing lining construction. (After Kalling, Josefsson, and Strömstad.)

or of scrap. The latter might appear dangerous with speeds of 30 r.p.m., but as Kalling points out the risk is less than might be expected, due to the scrap being buoyed up by the metal bath. The time on blast with the 30-ton vessel is approximately 40 minutes, tap to tap times of about an hour being achieved. The process is remarkable, in that an initially high phosphorus content can be reduced to as little as 0.025 per cent. while the carbon is still as high as 0.5 per cent. The slag removed part way through the heat is low in iron and being high in phosphorus can be used as a fertiliser. The thermal efficiency is relatively high, due to the fact that the carbon monoxide is largely burnt before leaving the vessel. The high lining temperature that would be expected from combustion of carbon monoxide with oxygen is avoided by rotation of the vessel, the exposed part being continually cooled by subsequent immersion in the metal bath. Oxygen consumptions are rather higher than with the LD, *viz.*, 2300–2500 cu. ft. per ton. In addition to the original plant at Domnarvet there are now two others, one at Oxelosund in Sweden and the other at Sollac in France. Four other plants—three in Great Britain and one in the United States—are under construction. The two vessels at Oxelosund have a capacity of

110 tons and are intended to operate with low phosphorus iron. Those at Sollac are larger—120 tons—but still have a blowing time of only 50 minutes.

(b) MATERIALS

Numerous refractories have been tried for Kaldo linings, but so far tar-bonded dolomite blocks appear to be the most economic. At Domnarvet the working lining is 14 in. thick and is backed by a 4½-in. safety lining of magnesite bricks, which is in turn separated from the shell by a 2-in. layer of insulating firebrick. Tests at Domnarvet showed imported dolomite to be superior to the local product, due it was thought to greater purity. Subsequent tests, in which fluxes equivalent to those present in the domestic material were added to the imported product, did not, however, yield any appreciable drop in lining life. It is now thought that high density rather than purity is the vital factor, a view supported by recent results on improved shaft kiln firing of domestic material. Residual carbon is also thought to be important, though additions of carbon to the lining batch do not appear to have given any appreciable benefit.

The vessel shown in fig. A13, p. 741, has a 6-in. (150-mm.) permanent lining of magnesite bricks and 15½-in. (400-mm.) thick working lining of pressed tarred dolomite bricks. These are manufactured from a blend of burned dolomite having the following grading, obtained by crushing, screening and grinding:

20–10 mm.	10 per cent.
10–7 mm.	30 per cent.
7–2 mm.	20 per cent.
<2 mm.	20 per cent.
<0.5 mm.	20 per cent.

The tar content is given as 6–7 per cent.

The 2-in. (50-mm.) gap between the two linings is rammed with tarred dolomite, giving to the brickwork the necessary rigidity. A filling of chrome-magnesite bricks is used at both ends of the vessel to provide the desired shape, while the furnace bottom (not inserted until the rest of the lining has been burned) is fully rammed and burned before installation. In more recent installations the insulating layer has been abandoned, apparently with a small gain in lining life.

The procedure adopted at Oxelosund is similar, except that Belgian dolomite is used. Incidentally it is interesting to note that in trials at Surahammar, using low phosphorus iron and a 10-ton silica lined



vessel, the refractory consumption was only 22 lb. (10 kg.) per ton—a tribute to the effectiveness of cooling by the molten metal.

(c) LIFE AND CAUSES OF FAILURE

The life given initially by Kalling for the working lining at Domnarvet was 50 heats, or about  $\frac{1}{4}$  in. wear per heat. The dolomite consumption was approximately 45 lb. per ton, corresponding to a magnesia content in the final slag of 4 per cent. The area of lining per ton of capacity is markedly less than that of the LD, and the working volume per ton only about one half. Both these factors might be expected to increase the wear rate in inches per minute.

Replacement of the dolomite fines by magnesite has yielded modest gains, while tar-bonded magnesite lining (90 per cent. MgO and 8 per cent. CaO on a tar-free basis) gives a life of 106 heats. Trials have also been made of fired magnesite bricks but these have so far given a maximum life of 93 heats. This relatively low figure is attributed to spalling, due, it is thought, to cracks formed in the slag-impregnated section of the lining. Magnesite bricks do, however, appear to be economic when used in the converter mouth, where temperatures are unusually high, due to lack of cooling by the molten bath.

Samples of dolomite lining from Domnarvet have been subjected to detailed examination by chemical, X-ray, and petrological methods. As a result it would appear that the iron oxide at the working face is present mainly as dicalcium ferrite, though in a more reduced condition further into the lining. Since large amounts of lime and iron ore are added at the start of the blow it is hardly surprising that their reaction product should figure strongly in refractory wear. Samples of used magnesite brick have also been examined and again show dicalcium ferrite at the working face and FeO in solution in the magnesia. Porosity measurements yielded figures as low as 2 per cent. for the hot face, compared with 17.5 per cent. further back. Assuming attack to be mainly due to dicalcium ferrite it is interesting to calculate the relative solubilities of dolomite and magnesite in such slags. Calculations made in 1958 by Messrs. Ford and White suggested that dolomite was likely to be three times as soluble as magnesite. The fact that magnesite only shows in practice a slight improvement over dolomite suggests that other factors, *e.g.*, spalling or stoning, are involved, or alternatively that the protective action of carbon mentioned in connection with the LD converter may be operating. In a 1962 paper, Kalling summarises the factors influencing attack as follows:

- (1) With a fluid slag rotation may cause increased attack by increasing the transport of active constituents through the boundary layer.

- (2) Heavy scrap may cause wear, though this is in part offset by buoyancy.
- (3) Continuous variations in the stress pattern due to rotation may cause spalling.
- (4) Excessive temperature rises may occur periodically, particularly in the nose of the vessel.

During a number of heats the slag was weighed and analysed and the dolomite dissolved calculated from a magnesia balance. The results suggest that the rate of solution of dolomite during the main part of the blow is only 25 per cent. of the overall rate, the worst damage occurring in the period when the slag is high in iron oxide.

The preliminary results on the 120-ton vessel at Sollac suggest lives of about 50 blows and a refractory consumption of 62.7 lb. (28.5 kg.) per ton. At Oxelosund the first campaign on the 110-ton vessel showed the average lives of 66 heats and a record of 79. With this low phosphorus iron the consumption of dolomite was down to 37.4 lb. (17 kg.) per ton of steel. Refractory costs, including labour, at Domnarvet are given as approximately 11s. 6d. per ton.

#### (d) LINES OF IMPROVEMENT

The best hope for a radical improvement in Kaldor linings would seem to lie in greater understanding of the mechanism of failure. Improvements are likely, however, to come from other directions, *e.g.*, by improving, say, by lance movement, the aerodynamic and hydrodynamic conditions. The progress already made suggests that the immediate improvement, however, is likely to come from tighter control of the tarred dolomite used, particularly as regards purity and density of the raw material. Closer control of the chemical reactions and the consequent temperature may also enable satisfactory steel to be made without the high iron oxide slags believed to be responsible for most of the damage. The use of computers for process control, as already employed by certain LD operators, may also lead to increased lining life.

## ROTOR

### (a) CONSTRUCTION

The rotor process, as used at Oberhausen and Pina in Germany, at Iscor in South Africa, and Richard Thomas & Baldwins Ltd., Redbourn, was first described by Graef in 1956. Fig. A15, p. 748, shows that the vessel is very different in shape from the LD and Kaldor, being essentially a horizontal refractory lined cylinder with a work-hole and two tapholes. The Oberhausen vessel before lining is 49 ft. (15 metres)

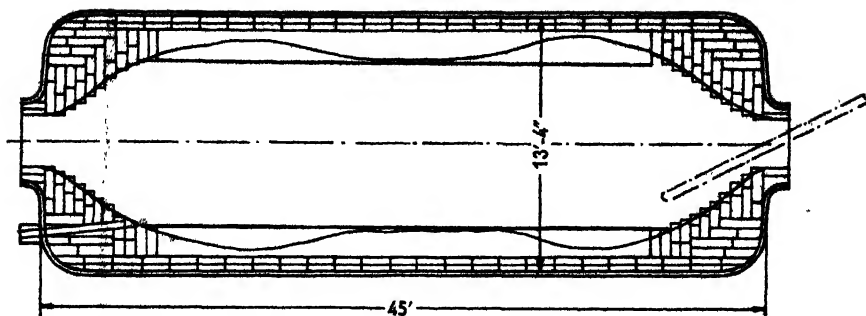


Fig. A15. Section through Oberhausen rotor showing wear lines. (After Spetzler.)

long and 13 ft. 4 in. (4.1 metres) diameter, and takes a heat of approximately 90 tons. A double-ended lance is employed, the primary section being immersed in the bath, while the secondary section provides oxygen above the bath to burn the CO liberated from it. Lime and other solids can be blown into the furnace through apertures in the secondary lance in order to control slag composition. The latest units are designed not only to be tilted but to be swivelled horizontally, so that lancing can be carried out at either end and wear spread more evenly throughout the vessel. Both the area of refractory exposed and the chamber volume are much larger than with the LD and Kaldo vessels. Rotation speeds are much slower than with the Kaldo, being originally only 0.1–0.5 r.p.m., compared with 30 r.p.m. for the Kaldo vessel. With such low speeds the value of rotation lies primarily in reducing refractory wear rather than promoting mixing. Without rotation extremely high temperatures would be obtained in the top of the lining due to the burning of hot carbon monoxide in oxygen.

#### (b) MATERIALS

The working lining of a rotor normally consists of tarred dolomite ramming, approximately 15 in. in thickness, with a 4½-in. safety lining of magnesite bricks. The end rings are built of chrome-magnesite, presumably because they must withstand both thermal shock and slags high in iron oxide.

#### (c) LIFE AND CAUSES OF FAILURE

In his original paper Graef pictures the conditions existing in the rotor, due to the boil and the combustion of the CO liberated from the bath. Under such severe conditions dolomite consumptions of 110 lb. per ton when making low carbon steels are hardly surprising. Tap to

tap times were originally about 2 hours but have been reduced to as little as an hour by increasing the blowing rate. The total consumption of oxygen for carbon reduction and combustion is given as about 3000 cu. ft. per ton. Lining lives under such conditions are only 3-4 days and a second vessel must, therefore, be installed in order to ensure continuity. This may prove difficult even with two vessels, since repair times may well be as much as 5 days.

In his 1962 paper Spetzler gives a much lower figure for refractory consumption, *viz.*, 28.6-35.2 lb. (13-16 kg.) per ton of crude steel, compared, he suggests, with 19.8 lb. (9 kg.) per ton for the LD process. Lives with high phosphorus irons in rotor vessels are given as 70-100 heats. It is suggested that longer lives could be achieved by avoiding complete combustion of CO liberated from the bath, since this would reduce the temperature to which the refractories are exposed. It would also of course reduce the thermal efficiency of the process. Numerous refractories have been tried in the various rotor plants but so far very little data have become available. Among the materials tried are tarred dolomite ramming, unburned tarred dolomite bricks, high-pressure moulded burnt dolomite brick, magnesite brick, and magnesite ramming. In these experiments the magnesia content of the slag has been used as an index of lining wear and figures as low as 3-5 per cent., compared with 6-8 per cent. in normal practice, have been achieved. Tarred dolomite blocks gave lives comparable with ramming, while the high-pressure moulded and burnt dolomite blocks stood proud of the ramming when used in panel trials. Complete brick linings showed signs of spalling at 40 heats but were said to give an overall improvement of some 30 per cent. Magnesite bricks also spalled, pieces of about 4 cm. thickness coming away and the final life being only 20-30 per cent. greater than with rammed dolomite linings. In one steelworks lives of up to 150 heats have been claimed for rammed magnesite linings.

Numerous attempts have been made to fettle rotor vessels, using, for example, burnt dolomite and slag, tarred dolomite and unburned dolomite with slag. A robust oil-oxygen burner has been used to promote sintering and lives of up to 8 heats obtained on burned in layers. The most economical practice appeared to be the use of unburned dolomite, though the actual life was shorter than with the burnt material. Fettleing terms are given as 45 minutes per heat and dolomite consumption 77-88 lb. (35-40 kg.) per ton. With such fettleing two campaigns of approximately 1500 heats were achieved. The practice is considered practicable where two adjacent rotors are available, one being fettled while the other is making steel. Even without fettleing lives of 150-200 heats will it is hoped ultimately be achieved with high phosphorus iron.

(d) LINES OF IMPROVEMENT

In spite of the large number of experiments done on materials, design, and operation, the rotor still presents a major problem to the refractories technologist—both from the point of view of costs and availability. Where, as at Redbourn, the rotor has mainly been used for desiliconising, refractory problems are by comparison negligible, and it may well be in this field that the rotor will prove most useful. Other advantages likely to increase life are changes in shape, in particular increasing diameter to length ratio, but whether these make the rotor competitive with the LDAC, OLP, and Kaldo processes remains to be seen.

The major and widespread changes still occurring as a result of the use of oxygen in steelmaking will inevitably result in the present review being out of date even before it is published. In many plants improvements will have occurred both in the materials used and the lives obtained. Nor has mention been made of numerous other problems arising from the use of oxygen, *e.g.*, in pre-refining. The application of oxygen in arc furnaces was referred to in Chapter XII of the original text. Statements made there still apply, though considerable improvements have occurred in sidewall construction, notably the use either of a belt of electro-cast chrome-magnesite brick above the bath, or of large dolomite blocks. The main difficulty still arises when oxygen is used in large quantities for the melting of stainless steel scrap. This results in exceedingly high bath temperatures and necessitates the use of rammed magnesite instead of dolomite bottoms.

In concluding this addendum the author would like to thank all those who have so generously contributed information and illustrations.





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